

The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

Volume 70 Number 5

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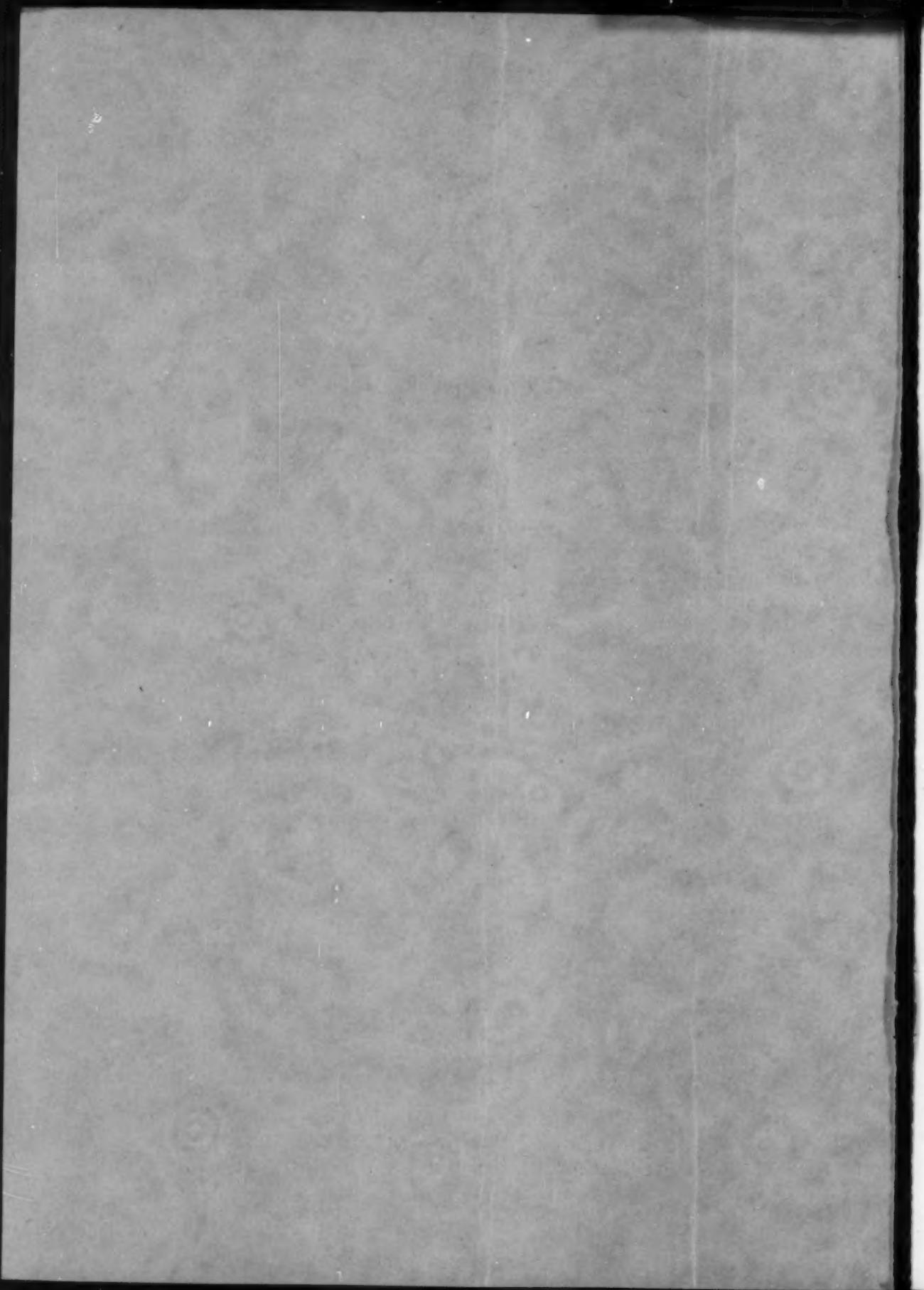
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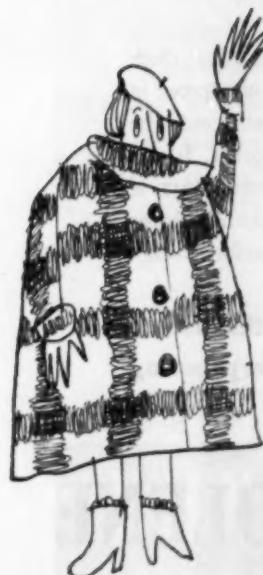


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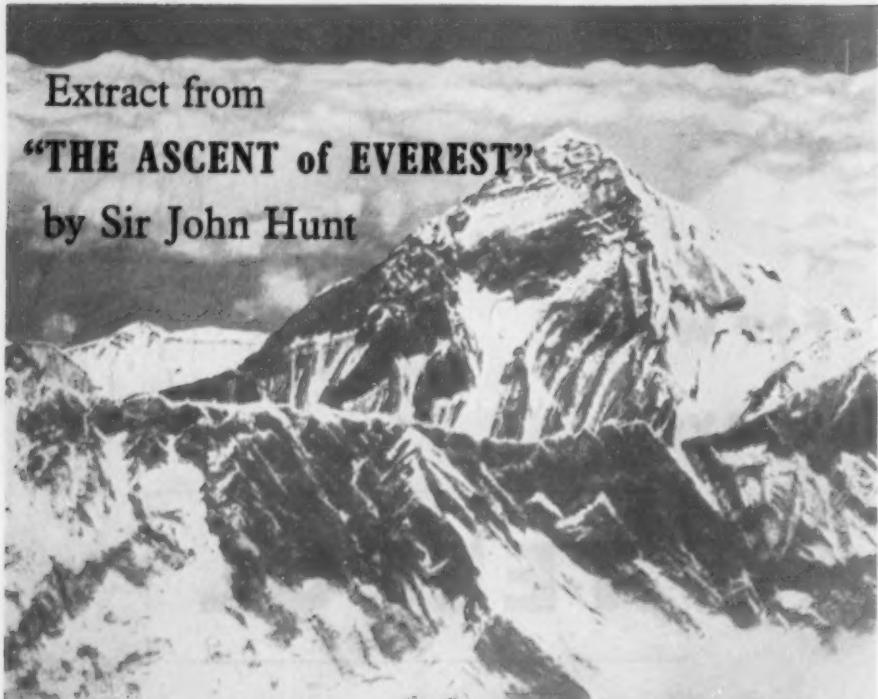
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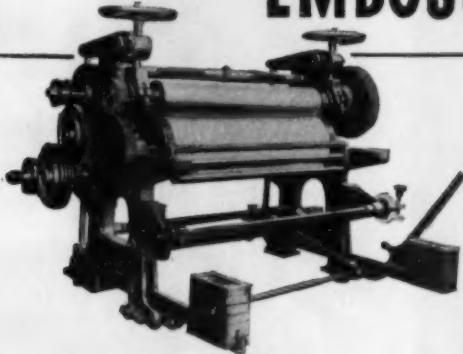
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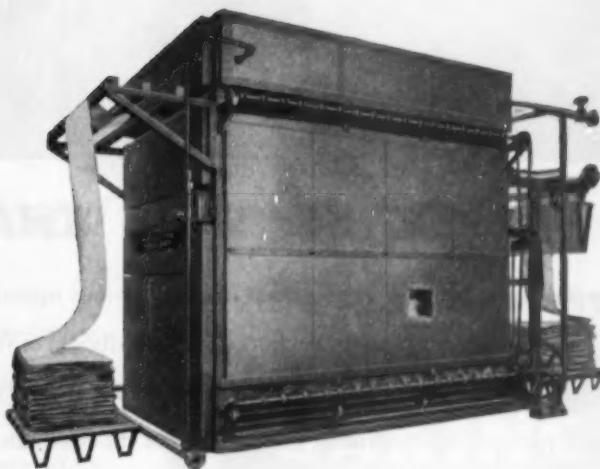
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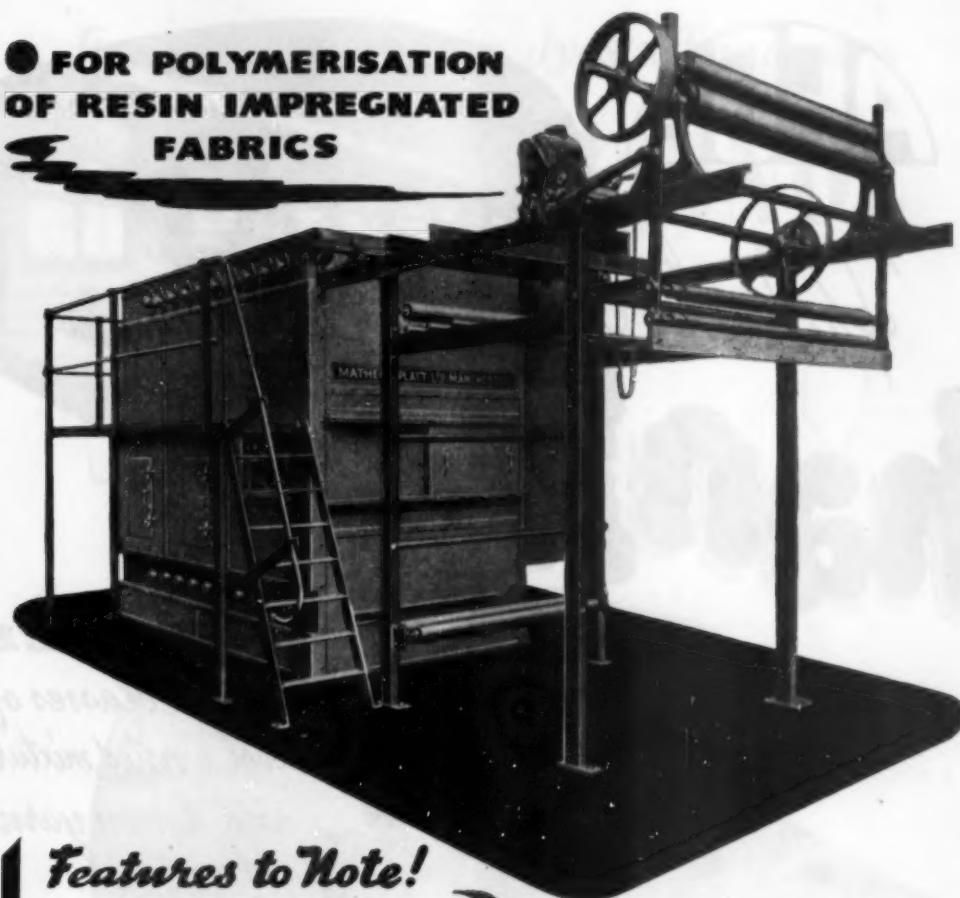
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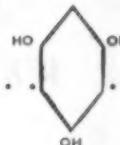
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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1954 and pages 229-232 of the July 1953 issues of the *Journal*, or write to *The Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138-9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

Fastness Properties for Users' Satisfaction	<i>J. S. Ingham</i>
Variables in Padding Processes	<i>R. W. Speke</i>

COMMUNICATIONS

The Polarography of Azo Dyes	<i>J. de O. Cabral and H. A. Turner</i>
The Preparation and Properties of some Inorganic Alginate Fibres	<i>M. J. Dudgeon, R. S. Thomas and F. N. Woodward</i>
The Combination of Wool with Acids	<i>L. Peters and J. B. Speakman</i>
A Laboratory Apparatus for Detergency Studies	<i>W. A. Straw</i>
Some Aspects of the Chemistry and Textile Uses of Sequestering Agents	<i>J. V. Summersgill</i>

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THE JOURNAL
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Proceedings of the Society

**Problems of the Garment Dyer and Finisher, including the
Dyeing of Buttons**

B. KRAMRISCH and A. E. BRATT

*Meeting of the London Section held in the Rooms of the Royal Society, Burlington House, London W.1,
on 6th November 1953, Mr. G. F. Wood in the chair*

The work of the garment dyer today is complicated by the advent of man-made fibres, which are used both alone and mixed with natural fibres. Moreover, the wide range of special finishes applied to textile materials destined for clothing and furnishings represents an added problem, as they are likely to interfere with dyeing processes.

In contrast to the textile dyer, who normally processes virgin materials, the garment dyer has to cope with materials whose dyeing properties have been altered by the action of light, perspiration, industrial atmospheres, washing, deodorants, and other toilet preparations.

In addition, garments may contain buttons and other decorations made from a wide range of natural and artificial plastic materials. They differ considerably in their physical and dyeing behaviour, so that wherever possible they should be removed from the garment prior to dyeing and if necessary dyed separately.

The garment dyer may also be called on to dye articles made from leather, furs, and sheepskins. This represents a special branch of the dyeing industry, and will not be covered in this paper. Consideration will, however, be given to the dyeing of soft furnishings and carpets, which are normally handled by most garment dyers.

Garment Dyeing

ALL-WOOL GARMENTS AND THOSE COMPOSED OF A UNION OF WOOL WITH COTTON OR CELLULOSE RAYONS

Very few garments fall into the first category, as, although garments may be ostensibly all-wool, they are likely to contain some cotton in the form of stitchings and buttonholes. All-wool garments are readily dyed with selected molecularly dispersed or aggregated wool dyes. Where cellulosic fibres are present, it is usual to employ the one-bath process with mixtures of aggregated wool dyes of the Cloth Fast type, along with direct dyes which

have minimum substantivity for wool from a Glauber's salt bath at 90-95°C. Certain direct dyes which give practically solid shades on the two fibres, such as Direct Scarlet BS and Chlorantine Fast Red K, are also used.

The action of light on wool has to be taken into consideration, and the coverage of faded wool is a complex problem.

The general tendency for acid wool dyes is to dye exposed wool lighter than unexposed wool. Neolan (premetallised) dyes, however, were found to give good coverage of faded wool and in some cases actually dyed the faded wool heavier than unfaded material¹. It was also established that this behaviour was to some extent due to the low pH used (1.9-2.1), as ordinary acid dyes applied at lower pH values than normal tended to show better coverage of fades. Neolan dyes are, however, of relatively little importance in garment dyeing, as, when cellulosic materials are present, neutralisation is necessary before the cellulosic fibre can be filled in from a second bath, whereas where possible a one-bath process is preferred on account of speed and ease of covering the cellulosic fibre. The newer premetallised dyes, Cibalans, are applicable at pH 6-7 and also cover faded wool very well, giving dyeings of high light and wet fastness, and so are of wider interest. They can also be used in combination with Chlorantine Fast and direct dyes in the one-bath process.

Several investigations have been carried out to ascertain whether the chemical constitution of dyes influences their covering power on faded wool. It has been claimed that a low degree of sulphonation of both acid and direct dyes tends to result in better coverage of faded wool². Further work indicates that, on the whole, dyes offer protection to wool against damage by exposure to light, but the degree of protection differs with the chemical constitution of the dye³. Triphenylmethane dyes

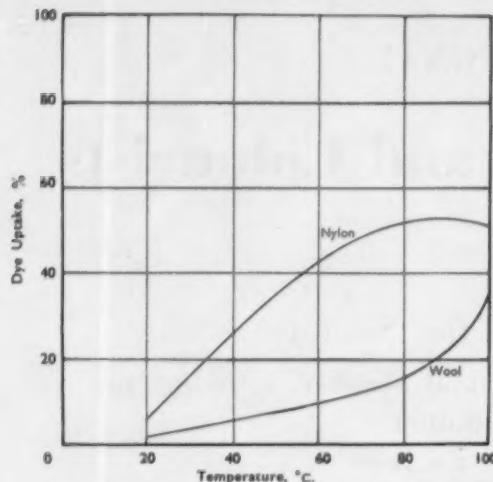


FIG. 1—3% Cloth Fast Blue RN on 50:50 Wool-Nylon

are stated to give the least protection, but this may be due to the comparatively low fastness to light of dyes of this type and their resulting decomposition on relatively short exposure.

Apart from the question of dye selection, the addition of 2-4 lb. Albatex PO per 100 gal. minimises the differences obtained on faded and unfaded wools, but the effect varies with the dye used.

GARMENTS COMPOSED OF MIXTURES OF WOOL AND POLYAMIDE FIBRES

A fundamental investigation of the dyeing of mixtures of wool and nylon has already been carried out⁴, and many of the recommendations made may be applied by the garment dyer.

Pale to medium dyeings can be produced with selected mixtures of acid dyes applied from a sulphuric acid-Glauber's salt bath.

In pale shades the nylon is dyed heavier than the wool, but this can be controlled by the addition of

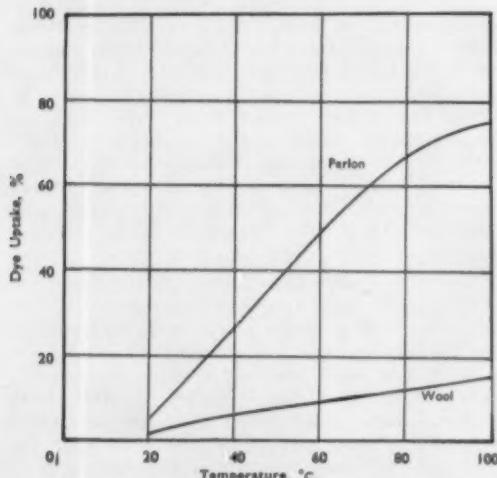


FIG. 2—3% Cloth Fast Blue RN on 50:50 Wool-Perlon

an anionic detergent, such as 0.5-2 pints Claytavon B per 100 gal. Full shades are best dyed with selected Cloth Fast, Acid Alizarine, or similar types of dyes from a neutral or weakly acid dye bath.

Caprolactam fibres such as Perlon and Grilon are also in use and differ in dyeing affinity from nylon, so that the relative dye uptake by the wool and polyamide fibre in mixtures differs according to the type of polyamide fibre used. A typical illustration is shown in Fig. 1-3, where the behaviour of 3.0% Cloth Fast Blue RN (C.I. 289) on 50:50 mixtures of wool and nylon, wool and Perlon, and wool and Grilon at temperatures up to the boil is illustrated. Cloth Fast Blue RN was selected as it is widely used for the production of fast navy dyeings on animal fibres. The polyamide fibres were all used in staple form (Table I), as they are normally blended with wool in this form. The wool in each case was 64s botany.

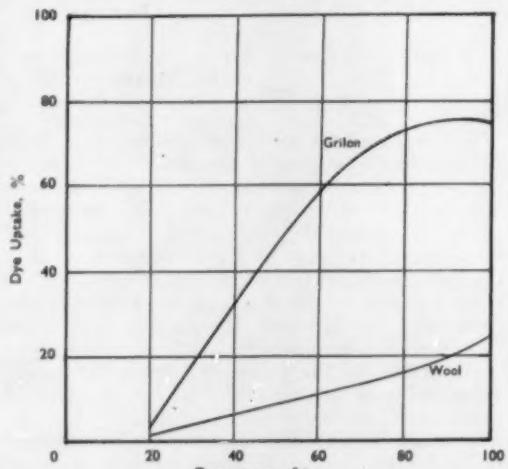


FIG. 3—3% Cloth Fast Blue RN on 50:50 Wool-Grilon

TABLE I

	Denier	Staple Length (in.)	pH of Aqueous Extract
Nylon	...	4	5.5
Perlon	...	4	6.9
Grilon	...	3½	4.2

It will be observed that, whilst a reasonably solid dyeing is produced on the mixture of wool and nylon, both the Perlon and the Grilon dye appreciably darker than the wool. It must be borne in mind, however, that some variation is likely to be obtained depending on the quality of polyamide fibre and its pH, and also on the proportion of wool and polyamide fibre present in the material.

GARMENTS COMPOSED WHOLLY OF NYLON OR OTHER SYNTHETIC FIBRES

Garments composed wholly of nylon or other polyamide fibres can be dyed with Cibacet dyes quite readily. The only difficulty arises if bright reds or oranges are required, as disperse dyes generally give bluer and duller dyeings on poly-

amide fibres than on acetate rayon. This difficulty can be overcome by the use of selected acid dyes such as Orange II and Acid Red S, provided the polyamide fibre does not tend to exhibit "barreness". Garments made of Terylene (ICI) polyester fibre can be dyed in a wide range of pale to medium colours by using the following selected disperse dyes applied without carriers—

Cibacet Yellow GBA Extra
 Cibacet Orange 2R
 Cibacet Orange 4R
 Cibacet Red E3B
 Cibacet Violet E2R
 Cibacet Sapphire Blue 4G

An addition of 0.5–1 lb. Albatec PO per litre to promote levelling is advisable. Carriers such as Tumescal D (ICI) will be needed for deeper colours.

It should be noted that the rate of dyeing of disperse dyes on Terylene is lower than that of the same dyes on acetate rayon. Dyeing takes place slowly at low temperatures, but the rate of dyeing increases as the boil is approached, and it is advisable to dye for 1–1½ hr. at the boil.

Vinyl polymer fibres such as Fibravyl, Rhovyl, and Thermovyl tend to shrink to an appreciable degree even well below the boil, and if possible the garment dyer is advised to avoid processing them. Dynel, a copolymer of polyvinyl chloride and acrylonitrile, however, is more stable and can be dyed with disperse dyes at the boil. Care has to be taken that a temperature of 130°C. is not exceeded in finishing, otherwise this fibre may be adversely affected. Acrylic fibres such as Orlon (DuP) present a relatively difficult proposition. Although a number of pale to medium colours can be produced with disperse dyes, full colours are difficult under normal dyeing conditions and carriers are of no value. The cuprous ion technique involving selected acid dyes, mainly monosulphonates, is rather involved.

Basic dyes and modified basic dyes applied from an acetic acid dyebath at the boil permit the production of a wide range of bright full colours on Orlon of good fastness to washing and reasonable fastness to light, the latter property being superior in some cases to that of the same dyes applied to natural fibres.

Dyeing at elevated temperatures (120–130°C.) simplifies the dyeing of polyester and acrylic fibres, but no suitable high-temperature pressure dyeing apparatus is available for the garment dyer, and it is not feasible to convert existing garment-dyeing machinery, i.e. open beck, oval or overhead paddle, steam and air circulating machine, or frame dyeing machines.

GARMENTS CONTAINING ACETATE RAYON, TERYLENE, ORLON, OR DYNEL AND WOOL

Mixtures of wool and acetate rayon are best dyed by the one-bath method using combinations of selected neutral-dyeing wool dyes which give minimum staining of acetate rayon together with disperse dyes. The latter type of dye stains wool to an appreciable degree in the initial stages of dyeing, but as the temperature approaches the boil, the disperse dye tends to migrate from the

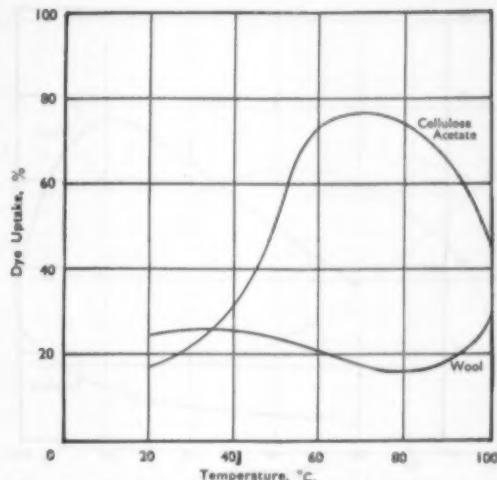


FIG. 4—2% Cibacet Orange 4R on 50:50 Wool-Cellulose Acetate

wool to the acetate rayon. However, even at the boil appreciable staining of the former fibre is shown, as will be seen by reference to Fig. 4, which illustrates the behaviour of 2% Cibacet Orange 4R on a 50:50 mixture of wool and delustrated cellulose acetate staple (6 denier, 6 in. staple) at different temperatures. Prolongation of the dyeing period at the boil tends to effect some improvement, however. Disperse dyes tend to rub on wool and also show appreciably lower fastness to light and washing on wool than on acetate rayon, so that it is advisable to restrain them from this fibre as far as possible.

A further point of interest is that dyeings prepared by the one-bath method exhibit better fastness to rubbing than similar combinations of dyes applied on the same mixture by the two-bath process.

Mixtures of wool with dynel, Terylene, or Orlon represent an even more difficult proposition. Fig. 5 illustrates the behaviour of Cibacet Orange 4R on

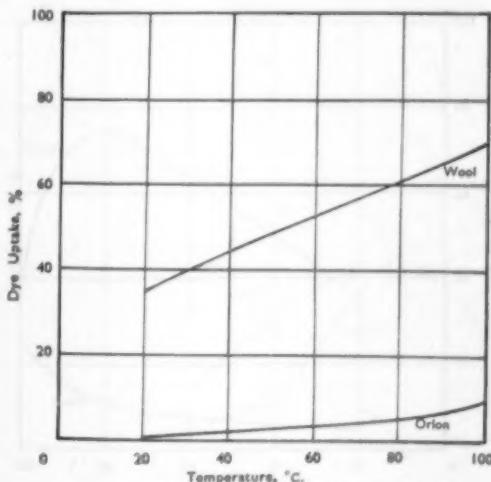


FIG. 5—2% Cibacet Orange 4R on 50:50 Wool-Orlon

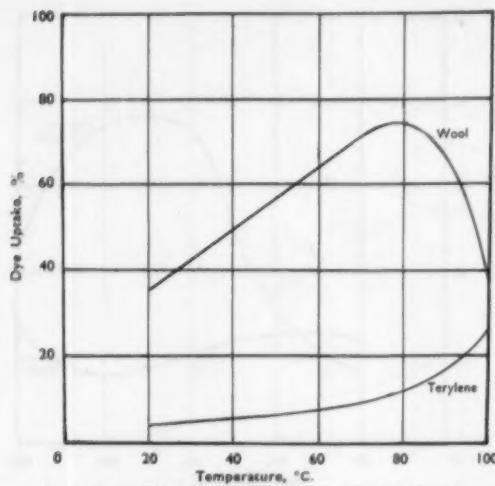


FIG. 6—2% Cibacet Orange 4R on 50:50 Wool-Terylene

a 50:50 mixture of wool and Orlon (4 denier, 2 in. staple), and indicates that even at the boil virtually all the dye is taken up by the wool. Fig. 6 and 7 deal with the same dye on 50:50 mixtures of wool and Terylene (3 denier, 2½ in. staple) and of wool and dynel (singles staple yarn, 20 denier). These two mixtures behave in a very similar manner: whilst at 75–80°C. the majority of the Cibacet dye is taken up by the wool, appreciable migration takes place on to the synthetic fibre at the boil, with the result that practically solid dyeings are produced on the two fibres. Although the situation is more advantageous with these two mixtures than with mixtures of wool and Orlon, the high percentage of disperse dye taken up by the wool and its low fastness to light, washing, and rubbing on this fibre make the dyeing of garments composed of these mixtures a very doubtful proposition.

The above remarks apply mainly to white or light-coloured garments and to a lesser degree to those in dark colours. The latter would require stripping prior to redyeing, but this represents an

extremely difficult proposition with garments composed of a mixture of wool and Terylene owing to the high resistance of dyed Terylene to hydro-sulphites, which are normally used for stripping wool. Although pure Terylene garments dyed with disperse dyes can be stripped fairly well with sodium chlorite, the use of this product has several disadvantages: it attacks stainless steels, is unpleasant to use, and is not to be recommended where Terylene is present with wool, as the latter fibre may be adversely affected. The situation with other synthetic fibres such as Orlon is similar.

GARMENTS CONTAINING REGENERATED PROTEIN FIBRES

In general regenerated protein fibres such as Ardil (ICI) and Fibrolane (Courtaulds) show a greater affinity for wool dyes than does wool at low temperatures, but this difference tends to disappear at the boil. Moreover, the wet fastness properties of dyes on these types of fibres are often inferior to those of the same dyes on wool. This property is particularly noticeable with the crystallloid type of dye, and therefore in deep colours it is advisable to select dyes from the aggregated types (Cloth Fast) in order to give satisfactory wet fastness properties. As far as possible Fibrolane should be processed at pH 4, and prolonged treatment at high temperatures should be avoided. These precautions are advisable in order to maintain the physical properties of this fibre. Wet cleaning of garments containing Fibrolane should not be carried out on the alkaline side, and a neutral detergent such as Claytavon B is suggested.

Both Ardil and Fibrolane tend to liberate formaldehyde in a hot dyebath, and this may exert a reducing effect on certain red levelling acid dyes such as Kiton Red G or Kiton Fast Red BG. This effect can be minimised by using an organic acid such as acetic acid in place of sulphuric acid or by employing Kiton Fast Red R or Kiton Fast Red 2BLE in place of the dyes mentioned. Formaldehyde may also be liberated during Hoffman pressing, and if absorbed by the press covers may affect other garments dyed with formaldehyde-sensitive dyes.

Providing the above points are taken into consideration, garments consisting of mixtures of wool and Ardil or wool and Fibrolane can be treated as all-wool.

A wide range of garments are made from mixtures of regenerated protein fibres with cotton or especially with viscose rayon staple. As Ardil may show an acid reaction, neutralisation of mixtures of this fibre with cellulosic fibres is advisable and treatment with 1-2 pints ammonia (sp. gr. 0.88) per 100 gal. liquor for 20 min. at 40–50°C. is usually satisfactory. Although it has just been stated that pure Fibrolane is best processed at pH 4, mixtures containing this fibre can be treated at pH 6 provided the time and the temperature of treatment are not excessive. Nevertheless, a useful precaution, after dyeing and washing off, is to give a final rinse at pH 4 to maintain the condition of the Fibrolane on subsequent drying.

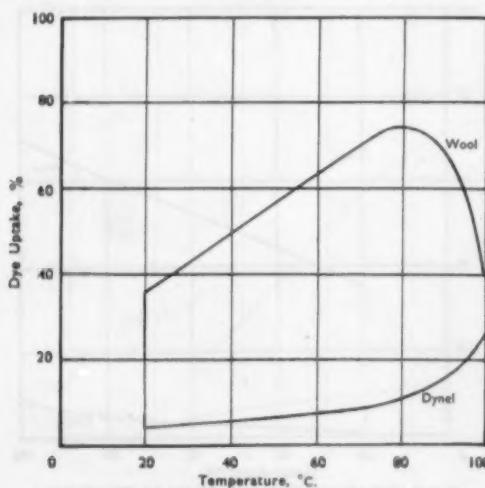


FIG. 7—2% Cibacet Orange 4R on 50:50 Wool-Dynel

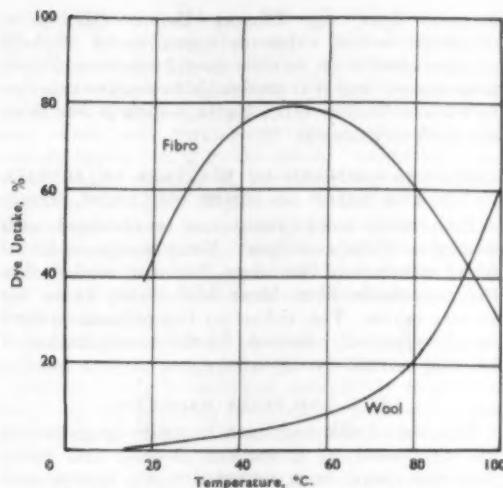


FIG. 8—2% Cotton Yellow CH on 50:50 Fibro-Wool

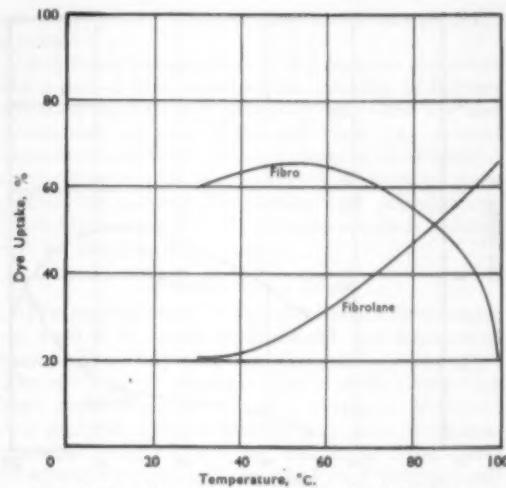


FIG. 10—2% Cotton Yellow CH on 50:50 Fibro-Fibrolane

Solid dyeings on mixtures of this type are obtainable with selected direct dyes under certain conditions; the uptake of dye by the regenerated protein fibre tends, however, to be greater than that of wool under similar conditions. This behaviour is exemplified by following the comparative uptake of 2% Cotton Yellow CH (*C.I. 365*) on 50:50 mixtures of Fibro (4½ denier, 2.5 in.) with wool, Ardil (15 denier, 4 in.), and Fibrolane (4 denier, 2 in.) respectively in Fig. 8-10. The effect will vary, of course, with the individual dye.

An alternative method is to use the one-bath union process, i.e. combinations of selected direct dyes and neutral-dyeing wool dyes as with unions of wool and cellulosic fibres. In the latter instance direct dyes are selected which give good reservation of wool from a neutral Glauber's-salt bath at the boil. With mixtures of regenerated protein fibres with cellulosic fibres, however, it is found that the former, in general, have greater affinity for such

direct dyes than has wool. The depth of dyeing on the two fibres can be controlled, however, by addition of Glauber's salt, as an increase in the amount of salt used results in an increase in depth on the cellulosic fibre, with corresponding decrease in depth on the regenerated protein fibre. An intimate knowledge of the behaviour of individual direct dyes is, however, essential, as marked differences are to be observed. For instance, Fig. 11, which illustrates the behaviour of 2% Chlorantine Fast Brown BRLL on 50:50 Fibro (4½ denier, 2.5 in.)-Fibrolane (4 denier, 2 in.), shows this dye to give solidity on the two fibres at about 75°C.; whilst Fig. 12, for 2% Chlorantine Fast Red K. on the same fibre mixture, shows solidity on the two fibres at 95°C.

The foregoing remarks apply principally to the redyeing of white or pale-coloured garments. With full colours there is the question of stripping, and hydrosulphite compounds will not give satisfactory stripping of disperse dyes on Terylene. Sodium

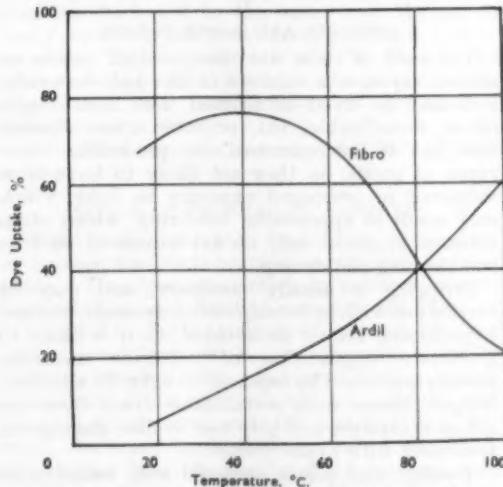


FIG. 9—2% Cotton Yellow CH on 50:50 Fibro-Ardil

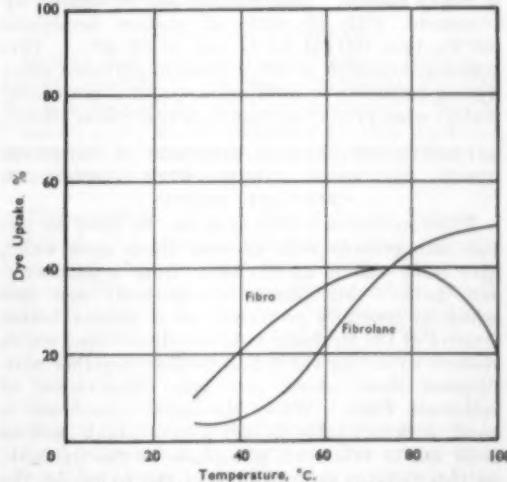


FIG. 11—2% Chlorantine Fast Brown BRLL on 50:50 Fibro-Fibrolane

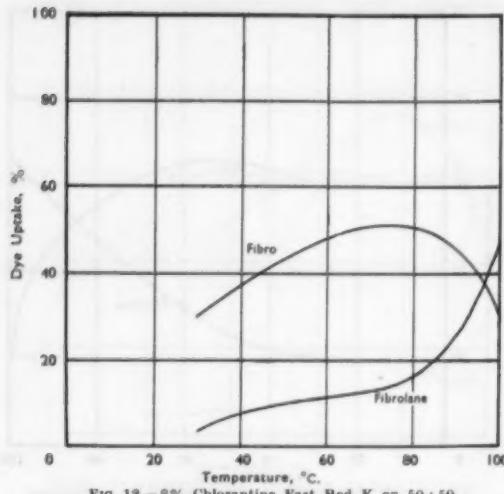


FIG. 12—2% Chlorantine Fast Red K on 50:50 Fibro-Fibrolane

chlorite is much more effective, although the use of stainless-steel vessels should be avoided, as the metal is liable to attack. In contrast, with unions containing wool, sodium chlorite can be used where cotton or viscose rayon is present. Sodium chlorite is also of value with fibres such as Orlon.

GARMENTS COMPOSED OF MIXTURES OF COTTON OR CELLULOSIC RAYON WITH ACETATE RAYON, TERYLENE, DYNEL, OR ORLON

These can be readily dyed to a wide range of solid colours by the one-bath process using combinations of direct dyes for the cellulosic fibre and disperse dyes for the acetate rayon or synthetic fibre. As already indicated, however, there are limitations to the depth obtainable on Terylene, dynel, and Orlon by this procedure.

Although there is an extensive range of direct dyes which will reserve acetate rayon and the types of synthetic fibres cited, even in full shades disperse dyes tend to stain cellulosic fibres when dyed in heavy shades. This staining can be cleared by treatment with 10 pints of sodium bisulphite (66°Tw.) per 100 gal. for 15 min. at 30–40°C. This staining, however, is not of great importance when dyeing garments to solid colours, and is noticeable mainly when producing reserve or two-colour effects.

GARMENTS COMPOSED OF MIXTURES OF NYLON OR OTHER POLYAMIDE FIBRES WITH COTTON OR CELLULOSIC RAYONS

Fibre mixtures of this type can be dyed by the one-bath process with selected direct dyes which give solid colours at 95–100°C. from a Glauber's salt bath. An alternative method, and one which is generally preferred, as it allows better control of the dyebath, is to use direct dyes which reserve nylon for the cellulosic fibre together with disperse dyes which give good reservation of cellulosic fibres. When the latter procedure is used, it is advisable to add a little alkali such as soda ash or trisodium phosphate to the dyebath, as this reduces any staining of the nylon by the direct dyes to a negligible amount. Individual

disperse dyes, e.g. Cibacet Yellow GN, show decreased colour value on nylon under slightly alkaline conditions, so that careful selection of dyes is necessary, and it is preferable to replace this dye by Cibacet Yellow GBA Extra, which is free from the fault mentioned.

GARMENTS COMPOSED OF MIXTURES OF ACETATE RAYON AND NYLON OR OTHER POLYAMIDE FIBRES

Reasonably solid dyeings can be obtained with a number of disperse dyes. Many orange, red, and violet members of the range, however, tend to dye the polyamide fibre bluer and duller than the acetate rayon. The colour on the polyamide fibre can be adjusted, however, by the incorporation of selected neutral-dyeing wool dyes.

SILK AND LINEN GARMENTS

The use of silk and linen in made-up garments has decreased to a marked degree, and these materials have been superseded by rayons and synthetic fibres. In general, silk can be treated as wool, and linen as cotton, as far as dyeing is concerned. Garments containing tin-weighted silk, however, have to be handled with special care, as the tin salts used in weighting may cause tendering of the fibre during wear, exposure to light, or storage, and this often makes itself apparent only during wet processing such as wet cleaning and dyeing.

RETINTING OF RAINCOATS

This process can be carried out in the dye vessel or preferably in the rotary washer, and by adopting the latter procedure raincoats can be retinted after wet cleaning, which obviates removal from the machines. Retinting is carried out with selected direct dyes applied without addition for 15 min. at 40°C. Better exhaustion of the bath can be obtained with 10% Glauber's salt, but this should not be added at the commencement of dyeing. Reproofing can be carried out after retinting by treatment with 10–20 lb. Migasol PJ per 100 gal. at 40°C., or alternatively the two processes can be carried out simultaneously.

CURTAINS AND LOOSE COVERS

The bulk of these are composed of cotton or viscose rayon or a mixture of the two materials, and may be dyed or printed with fast-to-light direct, metallisable, vat, or azoic dyes. Special care has to be exercised in processing these types of goods, as they are likely to have been subjected to prolonged exposure to light, which may result in appreciable tendering, which often becomes apparent only on wet treatment such as wet cleaning and dyeing.

Stripping is usually necessary, and may be carried out with hydrosulphite compounds. Sodium hypochlorite should be avoided, as it is likely to accelerate degradation of cellulosic materials already weakened by exposure to light. In addition, copper present from metallisable direct dyes can act as a catalyst and give rise to fibre damage on treatment with hypochlorites.

Dyeings and prints prepared with metallisable direct dyes should first be treated with 3–5 pints

of hydrochloric acid per 100 gal. for 30 min. at 50–60°C. to split up the copper lake, washed off, and then stripped with alkaline hydrosulphite.

Appreciable trouble was encountered at one time with vat dyeings and prints on cotton casements and other soft furnishings made of cotton or viscose rayon owing to the use of "tendering" vat dyes, which resulted in increased degradation on exposure to light. Extensive research work on this subject both in this country and abroad has led to the classification of vat dyes into "tenderers" and "non-tenderers", and most processors avoid the use of questionable dyes on materials intended for soft furnishings.

It will be appreciated that the majority of printed designs produced with the faster types of dyes can rarely be stripped completely to permit redyeing to a perfectly level colour, and even if the dye appears to have been destroyed, the printed design still tends to be obvious after redyeing.

Redyeing of soft furnishings is carried out with fast-to-light direct dyes (Chlorantine Fast dyes), or with metallisable direct dyes (Coprantine dyes) where improved wash-fastness is requested.

Curtains are also made from printed polyvinyl chloride sheeting and occasionally from printed glass fibre, but neither of these is capable of being redyed.

All-wool loose covers are dyed with acid levelling wool dyes, and those composed of mixtures of wool with other fibres are dyed by the appropriate dyeing method as already described for garments of this type.

CARPETS

Although the majority of carpets have an all-wool pile, the backing may consist of jute, cotton, or paper yarns. If the carpets are still in a good condition, it is necessary only to redye the pile with acid levelling dyes, preferably applied with an organic acid to preserve the backing. Wool-Ardil and wool-Fibrolane pile carpets can be processed on similar general lines to those containing an all-wool pile. If it is necessary to dye the backing as well as the pile of the aforementioned types of carpets, which will be the case where the pile is badly worn, the one-bath union process is recommended. This procedure is also to be adopted when the pile consists of a union of wool and cotton or wool and Fibro. All-Fibro carpets are best dyed with Class A direct dyes⁵.

A modified winch is recommended for redyeing carpets.

EFFECT OF FINISHES ON GARMENTS

Rapid strides have been made in the application of chemical finishes to textiles, particularly to piece goods. Therefore, many made-up garments manufactured from such finished materials are likely to be handled by the garment dyer, and this involves several considerations: e.g. whether the finish is likely to interfere with subsequent dyeing, and if so whether it can be removed in a simple and satisfactory manner, and the material then redyed. In many cases the removal of the finish may detract from the quality of the garment, and careful consideration should therefore be given to

all these points before a garment is accepted for dyeing.

A detailed examination of this problem is outside the scope of this paper, as the number of textile-finishing agents and patented processes for the production of special finishes runs into several thousands and involves both chemical modification of the fibre and the incorporation of chemical compounds of varying complexity and permanency. Therefore only some of the more common finishes will be considered.

Shrink-resisted Wool

The general effect of shrink-resistant treatments on wool is to result in decreased wet fastness of many types of dyes, mainly the acid levelling type. The effect on the aggregated dyes of the Cloth Fast type which are used in union dyeing is, however, less marked. The levelling properties of shrink-resistant wool often differ materially from those of untreated wool, and the effect may vary according to the type of treatment given⁶.

Embossed Finishes

These include finishes of the waffle, piqué, and Everglaze types, and are produced by embossing cotton materials which have been previously treated with a thermosetting resin, usually of the melamine-formaldehyde type. The permanency of the finish can be tested quite easily by wetting a small cutting taken from a seam of the garment. Slight tension or rubbing with the finger will indicate whether the design is likely to be removed. Where the resin treatment is permanent, the garments cannot be redyed, as the resin acts as a resist towards direct dyes.

Stripping of the resin can be effected by treatment with 3–5 pints of hydrochloric acid per 100 gal. for 30 min. at 50–60°C. followed by thorough washing off and neutralisation. If necessary, the treatment can be repeated. This treatment removes the embossed design and leaves a plain-weave fabric. Although it is possible to dye the stripped material, this is somewhat difficult because of modification of the cellulose.

Glazed Chintz Fabrics

Cotton materials are treated with a resin in a similar manner to embossed materials, and the finish is obtained by friction calendering. Similar remarks apply as for embossed finishes regarding examination for permanency of finish and its removal prior to dyeing.

Crease-resisted Viscose Rayon Staple Fabrics

Viscose rayon staple fabrics are made up into blouses, dresses, pyjamas, sports shirts, ladies' suitings and slacks, and soft furnishings, and are practically all crease-resisted with a thermosetting resin, usually of the urea-formaldehyde type. The finish is fast to washing and dry cleaning but is resistant to the majority of direct dyes. The resin can be stripped in a similar manner to that given for embossed finishes, and the stripped material can be redyed, although it no longer exhibits crease-resist properties. Crease-resisted rayon may liberate formaldehyde when garments are Hoffman-pressed.

This formaldehyde is then absorbed by the cotton covers of the press and may affect the colour of other garments dyed with sensitive dyes, e.g. certain acid levelling reds on wool. In such cases it is advisable to boil out the cotton covers of the presses when changing over from crease-resisted rayon staple to all-wool and union garments. Similar trouble may be encountered in pleating, as the cards used may contain a resin which liberates formaldehyde under practical pleating conditions; and also in the processing of garments containing Fibrolane or Ardil, which also may liberate formaldehyde under certain conditions.

Printed Seersucker Fabrics

Light-weight cotton materials are printed in stripes with thickened caustic soda solutions, or alternatively the material is printed in stripes with a resist, and the cloth is then passed through a strong caustic soda solution. The cloth shrinks where it has come into contact with the caustic soda and gives a "ripple" effect. No trouble is likely to be encountered in dry cleaning these materials, and on dyeing the treated portions may dye darker than the remainder, particularly in pale colours.

Retexturising Agents

These cover a wide variety of products including waxes and synthetic resins. Some are soluble in trichloroethylene and others in white spirit, and therefore the solvent used in cleaning will determine whether they are removed. If, however, this is not the case, they may exert a slight resist effect in subsequent dyeing.

The Dyeing of Buttons

At one time buttons were made from any available natural product which was of approximately the correct shape, and little thought was given to the colouring of these useful articles.

When people became more fashion-conscious and buttons were decorated, colouring was applied by painting, staining, and eventually by mordanting with inorganic salts followed by dyeing with vegetable extracts such as logwood, sumac, catechu, etc. The range of colours was very restricted and in most cases quite dull and lifeless; the results were uneven and difficult to control, while matching to pattern was almost impossible. In addition to these disadvantages it was found that the colour soon rubbed off, the colour changed considerably with ageing, and the colour lakes were susceptible even to mildly alkaline and acid conditions.

With the introduction of the synthetic dyes, brighter colours were obtained, and the method of dyeing was simplified, making pattern matching on a commercial scale a practical proposition. The first synthetic dyes—the basic dyes—were of poor fastness to light, and it was not until the wool dyes and vat dyes became available that really high standards of dyeing were attained. Synthetic dyes also have an advantage over the vegetable extracts, in that it is possible in most cases to remedy incorrect colours and faulty work by stripping and redyeing without damage to the button material. Originally all buttons were made from naturally

occurring materials, and many high-grade buttons are still made from these sources, but for cheap buttons the advent of modern synthetic materials has considerably increased the field of raw materials, and here as in many textile trades has necessitated new dyeing techniques.

BUTTON MATERIALS

Passing reference has been made to button raw materials, and for the purpose of this paper they will be grouped under four headings—

- I—Metals
- II—Vegetable
- III—Animal
- IV—Plastics

In the case of the first three groups the material is made by mechanical processing into buttons or button blanks, which are then dyed; but in the case of the plastic materials two alternative colouring methods are available—

- (a) Colouring the mass prior to moulding to shape
- (b) Moulding to shape and then dyeing.

DYEING AND FINISHING MACHINES

Except in the case of metal buttons, where each is treated almost individually, all the other materials are dyed by the following methods according to the bulk of work in hand—

- (i) In wire-mesh baskets immersed in the liquor.
- (ii) In inclined open tumbler drums, to facilitate movement and prevent the buttons from cohering into compact masses.

After being dyed, the articles are washed off, and are then dried on trays or in wire baskets at low temperatures (40–50°C.) by warm air. After being dried, the buttons are polished in closed tumbler drums, either with saw-dust and wax or in fine precipitated chalk, according to the material being coloured.

DYES AND DYEING METHODS

In view of the diverse materials to be considered, it is proposed to deal with them in groups according to their general characteristics, starting with the smallest group.

I—Metals

The use of anodised aluminium for the production of buttons is comparatively new, but this material offers considerable advantage over all other metals, except the precious metals such as gold and platinum, in that it does not require continual polishing. Moreover, in contrast with other metals used for buttons, it is cheap, can be produced in many surface forms, and above all can be dyed to any desired colour.

After being electropolished the buttons are anodised and then dyed with selected speciality products such as those known as Oxanal dyes. After being dyed at 70–75°C. for 10 min., the button shells are rinsed, and then treated in solutions of nickel or cobalt acetate followed by boiling water. Dyed aluminium buttons have other advantages in addition to those mentioned, since they are fast to dry cleaning, do not mark off

during steam treatments, and withstand mild washing conditions. Buttons of this class cannot be redyed without special pretreatments, and therefore require special apparatus not normally available to the button or garment dyers.

II—Vegetable Materials

The raw materials from vegetable sources are chiefly woods, although fruits such as corozo nut or vegetable ivory find quite extensive use. The woods are usually chosen for their natural beauty of grain structure, but quite an appreciable amount of wooden buttons made from walnut and ebony are not dyed. The woods most generally dyed are those of softer texture and somewhat more open grain, such as beech and boxwood, and the buttons are usually completely turned and drilled prior to dyeing. Buttons of this group can be either surface-stained or dyed, or less frequently completely penetrated, according to the dyes used and the time and the temperature of dyeing. Where bright surface colourings of no special fastness properties are desired, basic dyes are used from either aqueous or alcoholic media. If better fastness properties are desired, then speciality products such as the Orasol range of dyes in alcoholic solution are used. For surface dyeing in water medium, where fastness to wet processing is desired, then selected Chlorantine Fast and direct dyes are most suitable. If brighter colours and good penetration are the desired characteristics, with good to excellent light fastness, then the selection must be made from the acid or other series of wool dyes. Care must be taken to prevent excessive raising of the grain when wood is to be dyed, and this fault is aggravated by hot dye liquors. Unfortunately the dyer has often to weigh this disadvantage against reasonable penetration, since maximum colouring throughout the button is obtained only in hot dye solutions for long periods of time. This process is usually carried out at 80–90°C. for periods of 1–6 hr. according to the thickness of the button and the wood grain structure, but with very close-grain woods penetration is not possible without some form of high-pressure dyeing. After being dyed, the articles are rinsed, dried, and then polished.

With vegetable ivory considerable difficulties are encountered owing to the waxy nature of the material. Anything approaching reasonable results can be obtained only by careful and thorough pretreatment. This consists in immersion in cold water for 12–18 hr., with removal of the scum at regular intervals and constant working of the buttons. After steeping and swelling, the blanks must be thoroughly washed off under cold running water. Dyes are selected from the water-soluble ranges used for cellulosic and wool textiles, including basic dyes. Dyeing is carried out at 80–90°C. for 10–30 min. or until penetration is complete. Where maximum fastness to wet processing is desired and brightness is not an important factor, use is made of the Pyrogene (sulphur) dyes from the normal sulphide bath with the addition of Glauber's salt. In this case the washing off must be very efficient to remove all the sulphide odour.

III—Animal Materials

For animal buttons the manufacturer uses horn, hoof, bone, and ivory. The two latter forms are not widely met with today, and it is chiefly the horn and hoof of cattle, particularly Indian water-buffalo, that are used.

HORN—The horn material is thoroughly degreased by treatment with warm alkaline liquor and is then steeped in hot water to obtain a pliable material. This soft horn is then cut and pressed into flat sheets, from which the blanks are cut on special punching machines. It is also common practice to remove the horn tips and make these into dress buttons for ladies' wear. Horn buttons are rarely produced in bright mode shades, the important colours being grey, fawn, nigger browns, navy, and black. Since this is a quality material, the fastness properties are of a high order, and the dyes are selected from the Fournine or oxidation pigment class. The soft horn is first mordanted with selected inorganic salt solutions, such as ferrous sulphate, sodium or potassium dichromate, or copper sulphate, for 8–12 hr. at 30–40°C., and then thoroughly washed off in cold water.

Dyeing is carried out at room temperature for 2–3 hr., beginning in the solution of the Fournine for 30 min. To this bath is then added hydrogen peroxide, and the dyeing is allowed to proceed until the full colour develops. After being dyed, the material is rinsed, dried, and then drummed in hardwood sawdust. It is interesting to note that the pale shades of grey and fawn are not dyed direct, but are obtained by dyeing navy, black, or nigger brown and then machining off the surface until the less penetrated areas are exposed.

BONE AND IVORY—For bone and ivory, dyeing is carried out after degreasing, using selected acid and Neolan dyes, but where very bright colours are required, basic dyes are employed. All classes of dyes are applied at 60–70°C. for 30–60 min., and penetration is always poor.

SHELL—The raw material source in this case is known as *mother of pearl* and is composed of an animal exudation (*nacre*) on a background of calcium salts forming the shell proper. Owing to the presence of the calcium salts, most groups of water-soluble dyes are unsuitable for dyeing this material, since the tendency is to form a lake of poor fastness to rubbing and irregular distribution on the surface. The only satisfactory soluble dyes are basic dyes which do not form calcium lakes. The buttons are cleansed in cold ammonia solution and are then mordanted with sodium aluminate for several days if full colours are desired. Pastel tints can be obtained without mordant. Next the articles are rinsed and immersed in an alcohol-water (1:9) solution of the basic dye for several days at room temperature. The buttons are agitated from time to time, and are removed from the liquor when the desired depth is obtained. They are subsequently rinsed, dried, and polished in fine precipitated chalk.

Mother-of-pearl buttons are also subjected to inorganic lake formation to produce the "smoky" buttons of commerce. "Smoking" is the production of opalescent greys by silver salt formation, and

shades varying from brownish greys and blue-greys to almost black can be produced. The procedure is to clean the buttons and then immerse for one or more days in a solution of silver nitrate in a covered container. The articles are subsequently removed, rinsed, and then subjected to strong light, which "develops" the desired shade. Should deep colours be required, the treatment may be repeated several times. The variations in colour are obtained by development in weak sodium sulphide liquors.

CASEIN—This raw material is obtained from milk after the butter fats, sugars, and salts have been removed. Although it is animal in origin, its treatment during button formation closely follows that of moulding plastics. Casein plastic material is coloured in the mixing stages with pigments where maximum fastness to light, wet processing, and dry cleaning treatments is desired, and also with soluble dyes where the buttons are to be finally worked by turning on a lathe to produce two-colour effects. The classes of pigments used include inorganic, organic, and metal powders; and the soluble dyes are selected from the wool dye ranges. The greatest number of casein buttons, however, are made in the clear, i.e. no colouring matter is added, or are pigmented white with selected inorganic products such as titanium dioxide.

Casein stock is either formed in rods of varying cross-section or blanks are cut from sheets. When the stock has been formed, either by extrusion processes for the sections, or by hydraulic pressing for the sheets, the material is treated with formaldehyde for several days or weeks, usually about one day for each millimetre of thickness. Finally, the formalised material is rinsed, and carefully dried at low temperatures to minimise warping. Sections are next machined to size and then sliced, and sheets are pressed in heated moulds and the blanks subsequently punched out.

When dyeing is to be undertaken, selection of the colouring matter is of importance, and is made from the wool dye groups. The most important characteristics required are—

- (1) Fastness to light
- (2) Fastness to wet processing
- (3) Fastness to steam pressing

In order to improve the two latter characteristics, many dyes require special dyeing processes or aftertreatments, but as a rough guide to wet processing it is found that—

- (a) Ordinary acid wool dyes are the least fast.
- (b) Cloth Fast (aggregated) wool dyes are better, particularly so if aftertreated with salicylic acid
- (c) Neolan and Cibaland dyes (premetallised dyes)
- (d) Chrome dyes applied by the afterchrome method, and well washed off to remove free dichromate, yield the highest fastness to wet processing in deep colours such as blacks and navies
- (e) Maximum fastness of pastel mode shades is obtained by dyeing with Cibantines (solubilised vat dyes) and then "developing" with a persulphate and sulphuric acid.

Dyeing of all groups is carried out as hot as possible, usually at about 95°C., and is followed by washing off and drying under controlled conditions. The time factor is important, and dyeing may take anything from 1 to 10 min.; this is governed by the degree of formaldehyde treatment in the manufacture. If the material has been underformalised to allow for subsequent pressure working, the material dyes rapidly; if the buttons are completely formalised, then swelling is slow and the rate of dyeing is low. This variation in degree of formaldehyde penetration is often the cause of trouble on the larger buttons cut from thick stock or sheets, for both types of formalised casein are present in the cut area, and two different rates of dyeing occur, yielding unlevel results. When pattern matching is to be performed on precoloured blanks, the initial colour of the material must be taken into account, and this also applies to metal powder fillers, where, for instance, aluminium powder has a pronounced blueing effect.

IV—Plastics

Materials from this extensive group of new synthetic materials are becoming more and more common, and strong competitors to the older-established materials. It is possible with the wide choice available to imitate most of the natural products, with improved mechanical and other properties if the correct material is selected. Plastics are of two kinds—

- (1) Those which are moulded under heat and pressure, and are then resistant to further shaping; these are known as *thermosetting* products.
- (2) Materials which can be shaped by heat and pressure, and are still capable of further reshaping by heat and pressure; these are called *thermoplastics*.

In the first group we have phenol-formaldehyde, urea-formaldehyde, and melamine-formaldehyde, and these, subsequent to correct heating and pressure, become completely stabilised or cured, and cannot be redyed on the surface. These materials are therefore coloured in the powder or premoulding stage with selected dyes and pigments, and for the purpose of this paper are of little interest.

In the case of thermoplastics, many materials are used and quite a number of colouring operations are possible, but those of chief interest are—

- (1) Dyeing from organic-solvent solutions of dyes
- (2) Dyeing from aqueous medium with addition of swelling agents and wetting agents
- (3) Colouring in the mixing mass prior to moulding.

Buttons of this class are invariably moulded to shape; i.e. the button is in a salable condition prior to dyeing, either clear, translucent, or pigmented white. The most used products, of all the thermoplastics, for production of buttons, are—

- (1) Polymethyl methacrylate, a clear glass-like material
- (2) Cellulose acetate, clear or translucent

- (3) Polyvinyl chloride, for unbreakable buttons, rubber-like consistency
- (4) Nylon, opalescent white
- (5) Polystyrene, hard, clear, glass-like
- (6) Polyethylene, an opalescent waxy material.

The last two materials are hydrocarbons, and to date no satisfactory method of producing other than tints has been possible.

The other materials can all be dyed with reasonable success by the methods mentioned above. The dyes used are of the Cibacet (disperse) class, and are applied in a bath containing an organic swelling agent and a surface-wetting material along with water. Dyeing takes place at 80–90°C. from weak dyebaths for 5–10 min., and from strong dye solutions for as little as 15 sec., although this is not a recommended technique for reproducible results. Care must be taken not to use excessive amounts of swelling agent, or else surface crazing will occur and water will be absorbed into the surface giving undesirable "milky" effects, which cause rejects. After being dyed, the buttons are rinsed, dried, and then polished with either fine chalk or wood sawdust and wax polish.

The glass-like materials of this group lend themselves well to the production of high-quality decorative effects. Thus buttons may be moulded in two parts with an internal design which can be sprayed with solutions of Orasol dyes in a lacquer and vacuum-coated with aluminium, the back of the button then being fastened by an adhesive.

* * *

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THE CLAYTON DYESTUFFS CO. LTD.
MANCHESTER 11

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COMMUNICATIONS

A Laboratory Skein-dyeing Machine

W. ARMFIELD

A convenient laboratory dyeing machine is described for the dyeing of skeins. Dyeings can be carried out in liquor : yarn ratios as low as 10 : 1 using 5 g. of yarn. Twenty-four dyeings can be done simultaneously at the same temperature, or a maximum of twelve at each of two temperatures. Thermostatic temperature control is employed, together with continuous agitation of the yarn, which is kept completely submerged. The advantages compared with the usual pot dyeing methods are listed.

It is the custom in most dyeing laboratories to dye skeins of yarn a few grams in weight in dye pots, the skeins being suspended from V-shaped rods and turned periodically by hand to assist levelling. For many purposes satisfactory dyeings can be obtained by this method, but there are many disadvantages, particularly when working at low liquor ratios. The heating medium for dye pots is usually steam, hot air, water, salt solutions, or oil, but with any of these it is impossible to maintain constant temperatures below the boil in dye pots containing different volumes of liquor. Considerable temperature fluctuations are obtained when dyeing at high temperatures such as 90°C. at low liquor : yarn ratios. This is due to the cooling effect of the yarn, which is necessarily largely removed from the liquor during the turning operation.

With vat dyes, turning of the yarn increases the rate of oxidation of hydrosulphite, necessitating frequent testing of the dye liquors. Pot dyeing with vat dyes is particularly troublesome in this respect with low liquor : yarn ratios, and sometimes great difficulty in reproducing dyeings is experienced under these conditions.

There appeared, therefore, to be a real need for a dyeing machine which would overcome these difficulties, and which could be easily constructed

at a reasonable cost. Such a machine has been devised in which the yarn is dyed in narrow tubes, kept completely submerged, and agitated continuously; it is thermostatically controlled up to a maximum dyeing temperature of 95°C., and will allow 5-g. skeins to be dyed in liquor : yarn ratios down to 10 : 1. The machine consists of two units, which allow 24 dyeings to be carried out at the same temperature or up to 12 at each of two temperatures simultaneously.

The dyeing machine (Fig. 1 and 2) consists of two 16-gauge stainless-steel tanks 24 in. × 4 in. × 14½ in. high separated by ½ in. thickness of Insul wood. The tanks are similarly insulated on the outside and are finally enclosed in a wooden case. Water is used as the heating medium, and each tank is heated with a 1500-watt immersion heater, separately controlled by Sunvic TS2 thermoregulators and appropriate vacuum switches. A ½-h.p. motor with stirrer is mounted over one of the tanks, and a chain drive from this works a stirrer in the other tank. The motor speed is satisfactorily reduced by placing a 60-watt lamp in series in the circuit. A 52-r.p.m. Klaxon geared motor is connected to the rocker, which imparts a vertical reciprocating motion of 1½ in. to the skein holders (Fig. 3). The latter are made from stainless-steel rod (0.08 in. in diameter), and have an

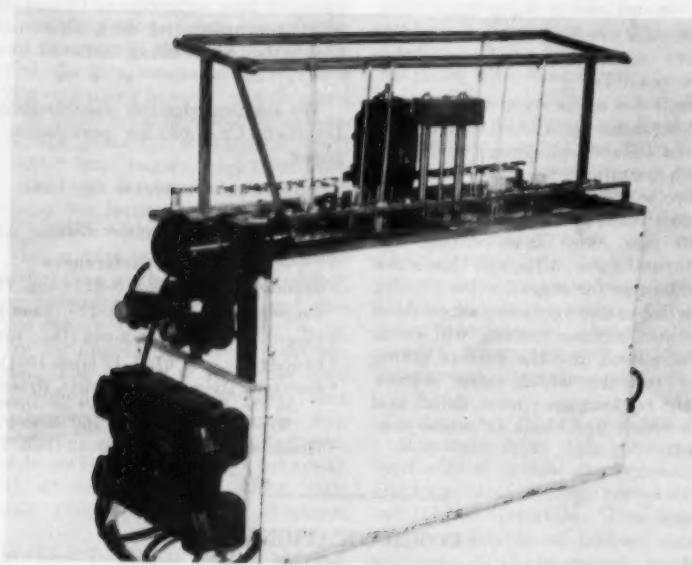


FIG. 1

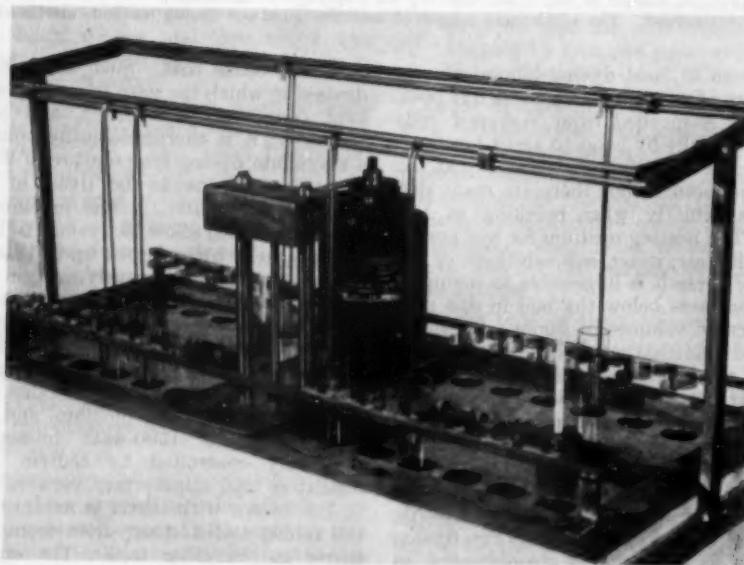


FIG. 2

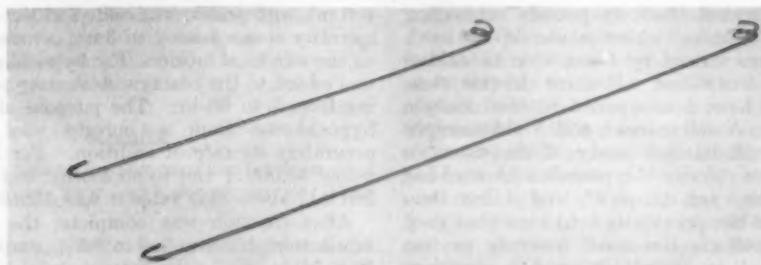


FIG. 3

overall length of 19½ in. and 12½ in. A 5-g. skein from a reel 43½ in. in circumference when folded to one-third of its length fits conveniently between the holder hooks. A top plate of 16-gauge stainless steel, drilled with holes not less than 1 in. in diameter through which pass the dyeing tubes, is fitted over each tank. The dye tubes consist of Pyrex glass tubing 14 in. long and of internal diameter 18-19 mm. for low liquor : yarn ratios, larger-diameter tubes being used where higher liquor : yarn ratios are required. The tubes are held in position by cadmium-plated Terry clips No. 80/3 mounted over the top plate. A perforated plate 3 in. from the bottom of each tank supports the tubes at the bottom.

The cost of materials is approx. £65.

A machine as described above has been used in this laboratory for several months, and the following merits and advantages are claimed for it compared with pot dyeing methods—

- (1) Completely satisfactory reproduction of dyeings
- (2) More even dyeings obtained with the difficult types of dyes.
- (3) All dyeings irrespective of volume of dye liquor can be maintained at any desired temperature up to 95°C.
- (4) The evaporation of the dye liquor during 30 min. at 95°C. is negligible.
- (5) The operator is free to carry out other work during the dyeing period.

(6) With the usual caustic soda and sodium hydrosulphite concentrations for vat dyes no testing of the dye liquor is required during a 30-min. dyeing period.

(7) The dyeing tubes are very much cheaper than dye pots.

(8) The machine occupies only a small amount of bench space considering the number of dyeings which can be carried out.

Although this machine was developed primarily for dyeing viscose rayon skeins with vat dyes in low liquor : yarn ratios, it has since been used with equal success for dyeing acetate rayon, cotton, nylon, and wool yarns. It is also specially useful for standardising dyes, and for dyeing laboratory matchings.

The idea of laboratory dyeing machines is of course not new, but it was thought worth while to make known this information in view of the reasonable cost of this machine, together with the special features mentioned, and the fact that it can be so easily constructed in a laboratory workshop.

* * *

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(Received 5th January 1954)

The Chlorination of Wool at Long Liquor Ratios*

C. EARLAND and D. J. RAVEN

It is shown that the effectiveness of chlorine in acid solution as an anti-felting agent for wool is independent of the liquor : wool ratio up to 100,000 : 1. It is considered that this is further evidence for concluding that the reaction between wool and chloramines is not a simple chlorination.

Introduction

The chlorination of wool as normally practised consists in exhausting on to the wool a definite weight of chlorine from aqueous solution. The volume of the latter, i.e. the ratio of liquor to wool, is not a critical factor in the reaction, and the choice of liquor ratio is usually one of convenience. It normally varies from the minimum necessary for

effective agitation of the wool to about 100 : 1. Over this range of liquor ratios no differences in the felting properties of wool treated with a given weight of chlorine have been reported, other factors such as the pH of the solutions remaining constant.

In order to overcome the major defect of chlorine as an anti-felting agent for wool, which is that its rapidity of action produces unlevel treatment, it

* Strictly, the wool was treated with chlorine in solutions of low concentration. Since for practical purposes this is equivalent to a long liquor ratio, to conform with textile practice, chlorine concentrations are given not only as true concentrations, e.g. e.g. of chlorine per 100 ml. of solution, but also as equivalent liquor ratios, which, since the wool was treated with 3.0% available chlorine, are given by the values of 8/8.

has been suggested that compounds containing "active" or "positive" chlorine should be used. These are characterised by being able to oxidise iodide ions to free iodine. Positive chlorine compounds which have been applied to wool include chloramine-T¹, *N*-chlorourea², and *N*-chlorosulphamic acid^{3,4}. A detailed study of the reactions of some positive chlorine compounds with wool has shown that they are complex⁴, and it has been concluded that the previously held view that they react with wool via the small quantity of free chlorine present in their solutions⁵ is incorrect. On the other hand, it has been established that certain positive bromine compounds, e.g. *N*-bromoacetamide and *N*-bromosuccinimide, react with wool largely through the intermediate formation of free bromine⁶.

Part of the evidence against the reaction between certain positive chlorine compounds and wool being a simple chlorination is that they can react with wool without producing resistance to milling shrinkage. *N*-Chlorourea and *N*-chlorosulphamic acid behave in such a manner. It has also been established that their solutions contain free chlorine. Thus a solution of *N*-chlorourea at pH 1.5 and 40°C. contains free chlorine at a concentration of 7.5×10^{-6} g.-mol./litre⁴, but wool treated in this solution is not rendered unshrinkable unless chloride ions are added.

Although, as has been previously pointed out⁴, it seems highly improbable on kinetic grounds alone that the reaction between *N*-chlorourea and wool could proceed via this extremely low concentration of free chlorine, which corresponds to a 3.0% chlorination at a liquor:wool ratio of 60,000:1, it appeared desirable to examine the anti-felting action of chlorine when applied to wool at liquor ratios of this magnitude.

It is known that chlorine attacks a number of sites in the wool structure, but it is unlikely that all are connected with the anti-shrink reaction. Thus, if the concentration of chlorine were reduced below that necessary to saturate all the reaction sites, reaction could occur at those unconnected with the anti-shrink reaction, and under these conditions the effectiveness of a given quantity of chlorine as an anti-felting agent would be reduced.

Experimental

1. CHLORINATIONS

The wool used was a two-fold 18s yarn of 56/58s quality knitted into fabric using 12 needles and 18 courses per inch, and cut into 5.0-g. single squares. Before use all samples were scoured in a warm dilute solution of soap and ammonia, and were then washed thoroughly in water.

A wet pattern was sewn on to a glass stirrer driven at 100 r.p.m. and entered into 800 ml. of 0.5% (wt./vol.) sulphuric acid solution contained in a one-litre round-bottomed flask, fitted with a double neck. Into one inlet was fitted the stirrer guide, and the tip of a burette was inserted into the other. By means of the burette, 2.0 ml. of commercial hypochlorite solution (7.50 g. available chlorine per 100 ml.), diluted to either 200 ml. or

400 ml. with water, was added to the wool. This quantity corresponded to 3.0% available chlorine on the weight of fabric. The hypochlorite solution was added to the reaction flask over periods varying from 1 to 60 hr. The purpose of adding the hypochlorite from a burette was to control accurately its rate of addition. For liquor ratios below 10,000:1 the hypochlorite was diluted to 200 ml., above this value it was diluted to 400 ml.

After reaction was complete, the patterns of fabric were hand-milled in 0.5% soap solution at 30°C.^{2,7}.

2. DETERMINATION OF APPARENT LIQUOR: WOOL RATIOS

The concentration of chlorine in solution during a chlorination is given in terms of apparent liquor:wool ratio, i.e. the liquor ratio at which it would be necessary to treat wool with 3.0% available chlorine to give the same concentration of chlorine in solution. Two methods were employed to determine the average concentration of chlorine present during chlorinations—

(A) A direct measure was obtained by iodometric titrations on 25-ml. portions of the solution, using 0.01 N. sodium thiosulphate solution. From four to ten such determinations, depending on the time of the treatment, were made. The solution removed for analysis had no significant effect on the degree of chlorination of the wool, and furthermore, since it roughly equalled that added from the burette, it did not affect the total volume of the reacting solution.

(B) A second method involved the calculation of this value from the velocity constant of the reaction. A pattern of fabric was chlorinated with the undiluted hypochlorite added in one portion. The progress of the reaction was followed by iodometric titrations on aliquot portions of the bath removed at definite intervals of time. The velocity constant *k* was calculated from the usual expression for a first-order reaction—

$$k = \frac{1}{t} \ln \frac{a}{a-x}$$

(*a* = initial concentration of chlorine and *x* = amount of chlorine changed in time *t*).

Since the concentration of chlorine in the solution remains approximately constant throughout the reaction—

$$\text{Rate of addition of chlorine to solution} = \text{Rate of reaction of chlorine with wool}$$

Since the volume of the bath remains approximately constant at 800 ml. throughout the reaction—

$$\text{Rate of addition of chlorine to} = \frac{0.15}{60T \times 8} \text{ g./min.}$$

(*T* = number of hours over which the chlorine is added). The rate of the reaction of the chlorine with wool is *k* [Cl₂]—

$$\therefore k [\text{Cl}_2] = \frac{0.15}{60T \times 8}$$

$$\text{or } [\text{Cl}_2] = \frac{0.000313}{kT}$$

where the concentration of chlorine [Cl₂] is expressed in g./100 ml.

These two methods for determining liquor ratios are completely independent, and agreement between the direct determination and the value calculated was reasonably good.

Results

The rate constant for the reaction between wool and chlorine at 20°C. was found to be 0.092 min.⁻¹.

In Table I are given the hand-milling shrinkages of patterns of fabric treated with 3.0% chlorine at different liquor ratios. It is seen that acid chlorine is an effective anti-felting agent at liquor ratios as high as 15,000:1.

Since a solution of *N*-chlorourea of pH 1.5 at 40°C. fails to render wool non-felting, although the free chlorine present corresponds to a liquor ratio of 60,000:1, there can be little doubt that the reaction which occurs with wool is not a simple chlorination. If it were so, there does not appear to be any valid reason why the fabric should not be rendered non-felting.

Further, the half-life of the reaction between *N*-chlorourea and wool is only 90 min., which is far too short if the rate-controlling step is the reaction between the free chlorine present and wool, since it has been shown in this investigation that the latter reaction under these conditions of extreme dilution is very slow.

TABLE I

Effect of Liquor Ratio on Shrinkage of Patterns treated with 3.0% Available Chlorine (pH 1.5)

Time of Addition of NaOCl soln. (T hr.)	Available Cl ₂ (g./100 ml.)—		Equivalent 3/A	Liquor Ratio 3/B	Area Shrinkage of Pattern (%)—	
	From (A)	From (B)			Treated	Untreated
TEMPERATURE = 20°C.						
0	—	—	160	—	8.5	33.8
1	0.00217	0.00340	1,400	880	4.8	33.8
2	0.00166	0.00170	1,800	1,800	7.0	33.8
5	0.00062	0.00068	4,800	4,400	7.2	33.8
16	0.00019	0.00021	15,800	14,300	7.4	31.4
TEMPERATURE = 40°C.						
0	—	—	160	—	8.5	31.4
1	0.00113	0.00171	2,700	1,800	10.0	31.4
3	0.00053	0.00057	5,700	5,300	7.6	31.4
10	0.00016	0.00017	18,800	17,600	9.6	35.2
60	—	0.000029	—	103,000	11.0	35.2

In view of the low rate of reaction between wool and chlorine at these dilutions, further experiments were performed at 40°C. in order to speed up the reaction. This temperature was chosen in order that the results would be comparable with those obtained from reactions between wool and chloramines^{2,4}.

The value of 0.183 min.⁻¹ was found for the velocity constant of the reaction between chlorine and wool at 40°C. Calculation of the energy of activation of the reaction from the velocity constants determined at 20°C and 40°C. gave the previously found value of 6 kg.cal. per mole⁵.

Results of the treatments performed at 40°C. also are summarised in Table I. Patterns treated at liquor ratios of 18,000:1 and 103,000:1 showed appreciable yellowing.

Discussion

This work has shown that the effectiveness of chlorine in acid solution as an anti-felting agent for wool is independent of the liquor ratio up to at least 100,000:1.

This investigation supports the previous conclusion that the free chlorine present in a solution of a chloramine, such as *N*-chlorourea, does not effect a simple chlorination of the wool, and its rôle will be discussed in detail in a forthcoming publication.

* * *

It is a pleasure to acknowledge the interest shown in this work by Dr. F. Happey, Head of the Department of Textile Industries.

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⁶ Earland and Raven, Text. Research J., 24, 108 (1954).

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⁸ Alexander, Gough, and Hudson, Trans. Faraday Soc., 45, 1058 (1949).

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Proposed Group for the Study of the Theory of Dyeing

It is customary to form groups within scientific societies to further specialised interests which, for geographical or other reasons, are not adequately catered for by the "normal" activities of the societies. Many such groups will readily come to mind.

It has occurred to the writer that the time may be opportune to form within our own Society a group for the study of the physical chemistry of dyeing in relation to the physical and chemical properties of fibres. A moment's reflection will convince even the most practical of dyers that progress in our industry will become even more dependent on fundamental research in the future than it is at present, and that such investigations should be fostered by all the means at our disposal. Such a group would not only serve this end but would also provide a useful means of disseminating valuable information.

The writer would be pleased to hear from all who are willing to give their active support to this project, and if the response is sufficiently encouraging a preliminary meeting will be arranged.

It is scarcely necessary to add that it is not intended to encroach in any way on Sectional activities, and that a group formed for the study of the physical chemistry of dyeing would meet with the approval of Council.

H. C. OLPIN

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(18th March 1954)

The Afterchrome Process

I have read with great interest Dr. Gaunt's paper "A Study of the Afterchrome Process" in the February 1954 *Journal*¹. From 1900 to 1920 I was in intimate contact from a dyemaking's angle with wool-dyeing circles. In my opinion Dr. Gaunt

overstresses the advance of the afterchrome process. I agree that it was quickly pre-eminent for the stock shades, blacks, blues, and browns, but I suggest that the chrome mordant process held its place for the fancy shades right up to 1914 owing to its greater simplicity for matching to shade. Anthracene Brown on chrome mordant was the kingpin for fancy shades until World War I cut off the supply.

The metachrome patent was dated 1900: it was quickly adopted by many wool dyers in Yorkshire: it was not adopted by the Scottish woollen or West of England dyers till World War I compelled its adoption for khaki uniform purposes. The West of England dyers continued the use of Alizarine Black WR and WX (B) for their London Black shade until the war cut off supplies in 1914.

In 1914 Metachrome Brown B was relatively freely available from British dyemakers. I recall that I had at the outset to provide technical service to many dyehouses which had hitherto not used the metachrome process. It was the technical changes enforced by the dye shortage in World War I which killed so quickly the chrome mordant process and Anthracene Brown with it.

It may be of interest to recall that the War Office specification for khaki uniform in 1914 involved the blending of five shades, whilst Anthracene Brown (MLB) was specified for the brown dye. It was my lot to have to write warning letters to all wool dyers using Read Holliday's Khaki Brown W (Metachrome Brown B) that its reactions were different from those of Anthracene Brown W when submitted to the tests laid down in the War Office specification. The pressure of events in 1914, of course, wiped all these away.

C. M. WHITTAKER

1 WEAPONNESS PARK
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(12th March 1954)

¹ Gaunt, J. F., J.S.D.C., 70, 46 (Feb. 1954).

New Books and Publications

Technical Manual and Year Book of the American Association of Textile Chemists and Colorists

Vol. 28 (1952). Pp. 643; Vol. 29 (1953). Pp. 673.
New York: Howes Publishing Co. Inc.

The format and the contents of these two volumes are similar to those of previous issues. As a yearbook this work is naturally mainly of interest only to members of the A.A.T.C.C., but as a technical manual it has proved itself invaluable to every textile laboratory throughout the world. In these volumes the list of journal articles commenced in previous issues is brought up to date, and a list of textbooks issued during 1930-1953 is

given. The lists of American-made dyes and auxiliary agents have also been brought up to date, and a number of new or revised test methods are included. It is indeed a manual which every tintorial technologist must have at his disposal.

C. O. CLARK

Veredler-Jahrbuch Deutscher Färberkalender 1954

(Volume 58)

Edited by O. Mecheels. Pp. v + 351 + 61. Munich: Franz Eder Verlag. Price, \$3.00.

The 1954 *Deutscher Färberkalender* contains, in addition to information on new dyes and auxiliary products manufactured in Germany and Switzerland

during 1953, a greater number of articles than ever before. These cannot fail to be of particular interest to members of this Society, as the majority deal with the development, application, and fastness properties of dyes.

The academic reader is catered for by articles on the effect of the purification of fibre and dye on dyeing, as well as on the development of dye synthesis and its relation to dyeing processes. The practical dyer will be particularly interested in such articles as those on machine dyeing; the dyeing of wool above 100°C.; developments in continuous dyeing; the colouring of polyacrylonitrile fibres, Dacron, and other synthetic fibres; the characteristics of Benzo Fast Copper dyes; and the application of Cuprophenyl dyes. The printer's interests have not been overlooked and there are a limited number of articles on natural and synthetic fibres, while the usual information on chemical compounds appears in the supplement.

The book cannot fail to be of the greatest interest to those of our readers who are familiar with German. It is unfortunate that this very useful information cannot be made available to a wider circle of readers in Britain and America by appearing also in English.

A. THOMSON

**Die Kupenfarbstoffe
und ihre Verwendung in der Färberel und
im Zeugdruck**

By F. Weiss. Pp. x+371. Vienna: Springer-Verlag
1953.

The book opens with a 40-page introduction by Dr. W. Reif on the chemistry of indigoid, anthraquinonoid, sulphur, and solubilised vat dyes, together with the theories of vatting, development, and the combination of vat dye and fibre.

Part I of the main work deals with the chemistry of the more important vat dyes. The constitutions of 35 indigoid and 133 anthraquinonoid and other vat dyes are given together with commercial names and references. Sections deal with the hydrolysis, over- and under-reduction, dehalogenation, relation between constitution, colour, and fastness of vat dyes, and the behaviour of leuco salts.

Part II consists of a very complete survey of the application of these dyes in dyeing and printing to a wide variety of fabrics including all types of cellulosic fibres, acetate rayon, wool, silk, polyamide and polyurethan fibres. Dyeing methods include, among others, padding, vat-acid, pad-steam, hot-oil, and molten-metal processes, while the chapters on printing cover direct, discharge, and reserve printing. The application of the solubilised vat dyes in dyeing and printing is also dealt with at great length.

It is a pity that a number of errors in the constitution of vat dyes which appeared in the original references should have been perpetuated, but these errors do not seriously interfere with the examples of the main types of vat dyes which the tables set out to show.

The book has an excellent list of contents, but would be improved by an index and, in particular, by a dye index, but, in spite of this, it is admirably planned, deals with the chemistry and application

of vat dyes in a very comprehensive manner, and should prove a standard book of reference to all students and users of vat dyes.

A. THOMSON

**The Identification of Textile Fibres
Qualitative and Quantitative Analysis of Fibre Blends**

By B. Luniak. Pp. xiv+177, +1 figure +14 tables + 312 photomicrographs. London: Sir Isaac Pitman & Sons Ltd., 1953. Price, 45s. Od.

This is the first English edition of a work of which there were two earlier German-language editions published in Zürich. The second of these was reviewed by the writer in 1949 (J.S.D.C., 65, 453). The new edition closely follows its predecessors, and it is sufficient to point out the changes which have been made.

There are no major changes, but there are two worth mentioning. One is that there are approximately 8% more photomicrographs, which extend the previously comprehensive collection to include recent man-made fibres. The other is the addition of a subject index. Besides these there are several minor changes, which do not require to be detailed but can be summed up by saying that they enlarge the scope and the usefulness of the work. Though the new English edition is well produced and larger than its predecessor, it is interesting to observe that the price is about 10% cheaper than that of the 1949 Swiss edition.

This supports the writer's impression that English publishing compares favourably with that of other nations.

The book can be recommended to those interested in this field.

J. M. PRESTON

Fiber Microscopy

A Textbook and Laboratory Manual

By A. N. J. Heyn. Pp. xiii + 410 + 12 plates. New York and London: Interscience Publishers. Price, \$5.50.

This textbook and laboratory manual is in a handy and compact form, and there is a wealth of information in its pages. Apart from the sections which are criticised below, the book is also successful in meeting the need of students for direction on the characteristics of different textile fibres and for methods of studying them in the laboratory. The scope of the book is wide, as will be seen from the twenty-two chapter headings, which are as follows—

- The Microscope and its Manipulation
- Preparation and Examination of the Unstained Fibre
- Cotton
- Flax and Hemp
- Ramie and Jute
- Mounting and Staining of Whole Fibres
- Micrometry
- Recording of Observations
- Wool and Other Animal Hair Fibres
- Hand Sectioning
- Rayons
- Resolving Power
- Silk
- Mechanical Sectioning
- Hard Fibres
- Paper Fibres
- Regenerated Protein and Alginate Fibres

Synthetic Fibres
 Mineral Fibres and Starches
 Fibre Identification
 Polarising Microscope: Investigation of Submicroscopic Structure [two chapters]
 Polarising Microscope (continued)
 Special Optical Methods.

There are several major criticisms which must be made. The section headed "Measurements of Fibre Diameters" (p. 97) receives scant treatment for such an important subject, and although the "wedge" method is briefly mentioned, the author recommends the old and tedious method which involves direct observation through the microscope and the use of an eyepiece micrometer; the quicker and better "profile" or projection method, which has been recommended by the International Wool Textile Organisation, should have been described. No mention is made of this method, nor of the instruments which have been widely used in this procedure, nor of sampling errors in the measurement of fibre diameter of wool.

Several sections of Chapter IX on "Wool and Other Animal Hair Fibres" are too superficial and even misleading. Only one type of medulla is described and other types known to exist are ignored. The generalised division of all mammalian fibres into beard hairs and wool hairs is not good and cannot be correctly applied to fibres from the sheep; this is evident from the author's superficial description of lustre wools as being ". . . almost entirely composed of beard hairs . . .".

The diagnostic criteria given for distinguishing animal textile fibres are frequently unreliable; the external scale patterns, which are so important an aid to fibre identification, are not mentioned. There are many incorrect descriptions, e.g. the statement that cross-sectional shape of the finer rabbit fibres is round. The complex scale-pattern shown by rabbit fur-fibres is not mentioned. The general inference made, when discussing the distinction between mohair and wool, that degree of scale overlap is correlated with the extent to which scales project from the fibre is incorrect: many wool fibres have prominent scale margins but very small overlap. The truth is that the degree of scale overlap is correlated with thickness of cuticle.

The accuracy of many other statements can be questioned, and the book fails to deal adequately with methods for the quantitative analysis of fibre mixtures (Chapter XIX) where animal textile fibres are concerned. It is stated that "Microscopic methods can be used for quantitative analysis only for the determination of the percentage of different fibre types by number". This is not true: the microprojection method, in which a length-biased sample is used and both number proportion of fibre types and their diameters are determined in order to provide a weight proportion, is reliable and is in regular use in some laboratories for the quantitative analysis of fibre mixtures.

The chapters concerned with the polarising microscope, resolving power, special optical methods, parts of the chapter on micrometry, and the chapter headed "Recording of Observations"

are excellent, and the directions for laboratory manipulations are clear and explicit.

There is a useful list of reagents near the end of the book and twelve plates of photomicrographs chiefly of the newer synthetic fibres. There is a good index. The book is well set out and provides a useful laboratory manual for the student of fibre microscopy provided its limitations, indicated above, are realised.

A. B. WILDMAN

Textile Prüfungen

Volume IB of "Handbuch für Textilingenieure und Textilpraktiker"

By P.-A. Koch and E. Wagner. Pp. xvi + 406. Wuppertal-Barmen: Dr. Spohr-Verlag, 1953. Price, DM. 26.00.

This book is divided into three parts—microscopy of fibres, physico-technological testing methods, and textile-chemical tests.

The physical testing methods comprise the greater part of the volume and are very comprehensive and thorough. All aspects of the physical properties are dealt with, and the text is amplified by many diagrams and tables. A special section of this part is devoted solely to comparative data for fibres and yarns, compiled in twenty tables. Much useful material will be found therein by the textile technologist.

Microscopy of fibres brings first a description of the microscope and its use, and then the methods for the identification of fibres, their appearance under the microscope, and finally special microscopic tests. This latter section is by no means comprehensive, and unnecessarily duplicates some of the subject-matter dealt with in the third part. This is all the more surprising as Parts One and Three are by the same author.

Part Three, textile-chemical tests, deals with the qualitative differentiation of fibres, quantitative determination of fibre constituents of mixtures, determination of impurities, tests for fibre damage, dye-chemical tests, determination of the pH value, and determination of finishes on the fibre. This part is concluded by a table concerning the removal of stains from fabrics.

Whilst the physical testing methods have been brought up to date by an appendix, no such attempt would appear to have been made in the other parts. Thus, much emphasis is still placed on the Allwörden test in the investigation of damage to wool, and on p. 58 of Part Three it is stated that "no special methods have so far been worked out for the investigation of damage to silk". This is probably partly due to the fact that mainly German sources of literature are considered.

The production is not entirely up to the standard of present-day German textile literature. The paper is poor and the print is not very clear. An attempt has probably been made to keep the cost down. All in all, this volume is a useful contribution to textile literature, especially insofar as it is part of a handbook consisting of many volumes dealing with all aspects of textile technology, and provides many interesting data.

E. KORNREICH

Wool Shrinkage and its Prevention
By R. W. Moncrieff. Pp. 576. London: National Trade Press, 1953. Price, 75s 0d.

The day has not yet arrived when this matter of felt-resisting wool can be dealt with in two paragraphs, as it ought to be eventually. Meanwhile, one is reminded of the poem of Elizabeth Wordsworth, how nice it would be "if the clever were always good and the good always clever". The good processes for curing wool felting are not particularly cheap, and the cheap processes are not very good.

The volume under review deals adequately and critically in great detail with the subject-matter up to the middle of 1952. Anyone who has to be *au fait* with the modern ideas and processes will find every aspect discussed. It should be made available to every library. It indeed fulfills what may be stated as a "non-felt want", so to speak. Perhaps it may also be described as the "Non-felt Bible", but even the Bible has its critics.

The book is entertaining and quite readable in an easy chair, the chapter headings being enlivened by classical quotations occasionally irrelevant but amusing. The most amusing of these is the final one from *Ecclesiastes VII*, which, one can imagine, was uttered by Mrs. Moncrieff, to whom the book is dedicated.

Since the work was published, several new cures have been published, e.g. the use of solid bleaching powder (Tootal Broadhurst Lee Co.), permmono-sulphuric acid (Stevensons (Dyers) Ltd.), and methylated methylolnylon (Lipson and Jackson). The last-mentioned has had some publicity in the popular press from scientific writers, writing in their experimental ignorance.

The author suffers at times from the advantage of apparently being a free lance without access to a laboratory, for he occasionally reads far more between the lines of a patent specification than is contained therein.

The book is singularly free from printers' errors, but the mechanism of polymerisation of methylated methylolmelamine on p. 387 must be corrected, for it is difficult to conceive the condensation proceeding as the author writes it.

The work is excellently printed and fully documented with references, but is too long, too heavy, and too dear. The next edition should be pruned and made more widely available.

F. C. WOOD

Manual on Industrial Water
ASTM Special Technical Publication 148
Prepared by A.S.T.M. Committee D-19 on
Industrial Water. Pp. 336. Philadelphia:
American Society for Testing Materials, 1953.

Water supply is one of the basic fundamentals which must be considered when siting an industrial plant, since practically every manufacturing process requires water in some amount or other. Water uses can be classed under two headings—industrial water and sanitary water. The first is used for all purposes not intimately connected with personal health. "Sanitary" water is used for food, beverages, lavatories, etc. Requirements for industrial water vary greatly: taste, colour, and

bacterial content are unimportant for, say, once-through condenser water, in which, however, scale- and corrosion-producing tendencies are of major importance. Colour, metallic impurities, etc. are, again, of more importance than bacterial purity for users in the textile and colour industries. The book under review is intended as a guide to executives and plant designers, those engaged in plant-processing which involves the use of water, analysts, engineers, and consultants, and it deals with the nature of water planning required at supervisory and investment levels and with the significance of the treatment which water undergoes. It accomplishes this object and also gives an excellent general picture of problems arising in many industries and the control and analytical methods employed.

Chapters are devoted to—difficulties caused by water in industry, composition of industrial water and water-formed deposits, treatment, sampling, and analysis of water and water-formed deposits. Both standard and tentative methods of sampling and analysis are given in the second part of the book, and a useful chapter is devoted to the methods of reporting results.

The book shows throughout the elaboration and punctilious attention to detail one has come to expect of publications of the American Society for Testing Materials. It forms a valuable compilation of knowledge on the industrial uses of water and problems arising from these and of the specific procedures for control, sampling, analysis, and reporting, together with useful constants, factors, and terms valuable to a water specialist. The book is probably too elaborate for the average dyehouse chemist's routine requirements, but occasions may be visualised when reference to this book may well prove most helpful and illuminating. The book is well illustrated and succeeds in giving, not only standardised methods of analysis, but also a good picture of the reasons for the desirability of following certain procedures.

The methods of analysis given will ensure results of stated accuracy and precision when used by an experienced analyst, and the alternative methods given are generally quicker and more applicable to routine work. Many of these alternatives are long established procedures in which extreme accuracy is a secondary consideration to speed.

A glossary of technical terms "not in common usage" completes the volume, but the reviewer feels that most of the terms are well known to the average chemist and could well be omitted, leaving the few more specialised definitions, peculiar to water-engineers, to be inserted in appropriate places in the text. This is, however, a very minor criticism of an excellent text.

J. A. RADLEY

The Chemical Analysis of Waters, Boller- and Feed-waters, Sewage, and Effluents
By Denis Dickinson. 2nd edition 1949. Pp. xii + 144. London and Glasgow: Blackie & Son Ltd. Price, 7s. 6d.

This is a useful little book containing a critical selection of established methods of analysis and examination of waters, sewage, and effluents,

together with practical discussion on the value of the results obtained. In chapters devoted to the separate subjects listed, the author discusses—the determination and significance of the physical characteristics; solids; alkalinity, acidity, and pH; hardness; ammonia; oxygen-consumed tests for organic matter; the biochemical oxygen demand; nitrites and nitrates; "heavy" metals; alkaline-earth and alkali metals; residual chlorine, salts, fluorine, silica; sewage effluents, grease, fats; boiler-feed and boiler waters; and the expression of the results. Throughout the book the author has expressed the results as parts per million, but has pointed out that this is a personal preference, since at present there is no universal system in use.

The absence of lengthy theoretical comparisons between various methods makes the book very readable and extremely handy for reference in the laboratory, and the inclusion of all the methods of expressing the results obtained is most helpful. The instrumentation necessary to carry out the various determinations has been kept to a minimum, and the Tintometer and a galvanometer are the most elaborate pieces of purchased apparatus suggested. The methods of carrying out the various determinations are clearly set out and concisely stated, and in many places the author gives useful hints

on practical manipulative details, which enable those using the methods for the first time to avoid the occasional pitfall. Several of the methods described for specific components of various waters have been elaborated by the author, and where recent work has vitiated a method described in the 1944 edition, the text has been amended to give the author's present choice.

One hopes that in the next edition the author will have something to say on the examination of swimming pool waters and on the determination of tar acids and phenols in water. Such additions would make the book complete as a water laboratory handbook. As it is, the book is a compact and valuable manual worthy of a place in every laboratory dealing with the examination of waters and their suitability for the many purposes for which they are used. It is particularly recommended to those who are called upon only occasionally to examine water for a specific purpose and, therefore, whose acquaintance with this subject is somewhat desultory. This by no means implies that those engaged in routine examination will not find much of interest in its pages, since the opposite is the case. It is adequately indexed and the price is most reasonable.

J. A. RADLEY

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

I—PLANT; MACHINERY; BUILDINGS

Future of the Jigger. Anon. *Dyer*, 111, 47-48 (8 Jan. 1954).

The use of the jigger in comparison with more modern dyeing machines is discussed. It is considered that this machine will diminish in importance but will nevertheless be necessary for certain styles.

A. H.

PATENTS

Driers and other Machines in which Fluid is projected into Fabric from Nozzles. J. Dungler. *BP* 703,243

If the fabric stops moving through the machine, shutters or other obturating means are automatically interposed between the fabric and the nozzles. This protects the fabric from the action of the hot air or other fluid without need for stopping the blower or pump and so enables stoppage and resumption of the treatment to take place in a minimum of time.

BP 703,391

Most efficient drying or other treatment is obtained when the distance between the fabric and the nozzles is $\frac{1}{2}$ 10 times the ratio between the sum of the areas of the blowing orifices of one and the same nozzle member and the length of the nozzle member over which the blowing orifices extend; in addition the sum of the cross-sectional areas of all the withdrawal passages placed between the nozzle members of each group of nozzles is not less than twice the sum of the areas of the blowing orifices of all the nozzle members in that group.

BP 703,392

The treating fluid is superheated steam, the rate of its flow and its temperature being controlled so that the liquid in the fabric is brought immediately to b.p. and is kept there during the passage of the fabric through the machine.

BP 703,556

The nozzle assemblies are so arranged that at least the main parts of the discharge currents are not directed transversely to the fabric close to its surface. This prevents lateral displacement of the fabric.

C. O. C.

Carrying Fabric through a Drying Machine. Aktiebolaget Svenska Flaktfabriken. *BP* 703,550

Conveyors for the pins of a tenter or similar machine are described which are light, occupy little space, and do not cause much loss of heat.

C. O. C.

Drying Liquid Coatings on Paper and the like. Butterworth & Furnival. *BP* 702,792

A machine for drying liquid coatings on paper, of the chamber and counter air flow type, and having inlets and outlets at the ends, has within it and wholly above the paper and at the top of the chamber a heat reflector and radiation screen below which are transverse heating elements. Between the screen and the heating elements are perforated deflectors, the whole being above a wire screen, below which the paper is passed with its coated side uppermost, the heat from the heating elements being reflected from the top of the chamber on to the deflectors, whereby air is heated and hot air and heat are deflected on to the coating.

S. V. S.

Selvedge Straighteners for Wool Fabrics. D. Foxwell. *BP* 704,156

A scroll device for uncurling the selvedge is followed immediately by a hot iron plate, against which the selvedge is pressed.

C. O. C.

Discharging Static Electricity from Sheet Material. Isotope Developments. *BP* 705,255

In a machine in which sheet material is being treated static electricity in the material is removed by connecting a radioactive source to the framework of the machine.

C. O. C.

Removing Carbonised Material from Cloth. H. B. Rand and R. N. Hoyt. *USP* 2,633,588

A crusher in which the cloth is treated in open width and time of treatment is shortened. It includes a set or sets of jaws through which the cloth passes and which vibrate 600-700 times per min.

C. O. C.

Machine for Printing or Coating Fabrics. A. J. C. de Oliveira Barros. *BP* 703,887

The machine comprises a pressure cylinder and several printing or coating rollers. The surfaces of these rollers are wholly permeable or permeable in parts to a colouring agent which is fed internally into the rollers. These rollers are applied against the pressure cylinder around which the fabric passes.

C. O. C.

Screen Printing. P. Langseth. *BP* 702,831

A printing table is used which consists of two or more plane surfaces symmetrically arranged about the axis of a rotatably mounted body. Rotation of the table brings a fresh portion of the fabric in position for printing.

C. O. C.

Drum Washing Machines. Machinefabriek Reineveld.*BP* 703,124

The perforated drum is radially divided into longitudinal compartments each of which is covered on the outside by two hoods extending circumferentially in opposite directions. These hoods are shaped so that (a) the goods are carried part of the way round the machine and then drop to the bottom of the compartment; (b) the exhausted liquor from the goods flows back into the casing; (c) fresh liquor is carried up from the bottom of the casing and is introduced to the goods as they pass the top of the machine.

C. O. C.

Rotary Electric Switch for Automatic Control of Laundry Washing Machines and the like. Baker Perkins.*BP* 704,670**Printing Ribbons to resist Laundering.** Milford-Astor.*BP* 703,496

The ribbon is passed over a table moved into contact with a heated die which applies a thermoplastic colouring material. On leaving the die it passes over guide rollers and over a stationary heated drum, so causing the colouring material to penetrate through the ribbon.

C. O. C.

Shrinking Hat Bats. United States Hat Machinery Corp. *USP* 2,632,937**Sequence Control Device for the Timed Control of One or More Operations on a Travelling Article.** American Laundry Machinery Co. *BP* 705,069**Glossing Paper.** U.S. Rubber Co. *BP* 704,394

A machine for continuously glossing paper comprises, a first roll having a hard non-resilient polished, e.g. mirror-polished chromium-plated surface, means for heating said roll, a second roll of diameter < 1.25 times that of the first roll, of highly compressed cellulosic fibre having a resilient surface, which is parallel to and arranged in conjunction with the first roll to form a nip for engaging a coated paper web, a third roll adapted to drive the second roll by friction; the first and third rolls are positively driven at equal peripheral speed, e.g. by a geared band connecting them, and the drive of the third roll includes a slippable clutch the drag of which is less than the lowest drag occurring in the nip of the first and second rolls when they are glazing, so that the peripheral speeds of all three rolls are equal regardless of the positive driving speeds of the first and third rolls. A coated paper web is continuously fed into the nip of the first and second rolls with the coated side adjacent to the first roll and lapped for about 180° to produce a plating-type surface gloss on the paper by the non-slipping contact of the first roll, and means, e.g. a fourth, parallel roll, for continuously drawing off the web tangentially from the first roll and in a straight line to maintain the gloss described. The first roll is heated internally to bring an unglossed coating of thermoplastic material on paper in contact with its surface above its softening point and convert it to a continuous highly glazed coating. The second roll may be positively driven separately, the clutch being interposed between the two drives, and the third roll is far enough from the first to allow the hot-glossed coating to cool below its softening point and permit reverse bending of the glossed paper without injuring the gloss.

S. V. S.

Plastic-coated Rollers. H. G. Lievremont. *BP* 705,298

A trued metal cylindrical core having gripping portions on its surface, e.g. studs, screw threads, or other protrusions

or recessed portions, and precoated from a tank with collodion, cellulose acetate, acetobutyrate or other material, is held between two centres connected with each other and moving in line with each other on a table or bench, the core thus being caused to pass through a box containing plastic coating material, e.g. a polymerisation product of vinyl chloride, which is subjected to a pressure ensuring its extrusion through an annular aperture provided around the core, the driving of which, with the two centres, is caused by the coating material extruded from the box and adhering to the metal core. The bench is provided with a track for aligning the centres and the box has an inlet diameter equal to that of the core and a larger diameter on the outlet side to regulate the thickness of coating applied. A heating device is placed at the inlet to the box and a cooling device at the outlet.

S. V. S.

Drying by Radiant Heat in vacuo of Material on a Moving Belt. F. B. *BP* 704,131

Modification of *BP* 696,655 (J.S.D.C., 69, 453 (1953)), an improved arrangement of the radiating tubes being described which prevents scale dropping into the material.

C. O. C.

Drying by Induction Heating of Colours, Lacquers, etc. on Metal Bodies. Daimler-Benz. *BP* 705,294**Automatic Valve Control for Washing, Bleaching, Dyeing, etc. Machines.** British Launderers' Research Association. *BP* 703,624**Coating Blade.** United States Rubber Co.*USP* 2,632,422

A coating blade which can be readily cleaned without dismantling the rest of the machine consists of a slotted tube which slides on a mounting plate passing through the slot, and in frictional engagement with the edges of the slot, into the interior of the tube.

C. O. C.

Felting Machine. Failsworth Hats. *BP* 704,078

In a machine for felting hat bodies in which the rollers are arranged in two horizontal rows more efficient felting is obtained if the rollers in one bank are helically threaded or grooved while those in the other bank are either plain or have separate endless grooves.

C. O. C.

Dyeing Textiles (VIII p. 209).**Printing Screens (IX p. 210).****Screen Printing with Resinous or Thermoplastic Colours (IX p. 210).****II—WATER AND EFFLUENTS****PATENTS****Disposal of Caustic Waste Liquor.** Allied Chemical & Dye Corp. *USP* 2,632,732

Strongly caustic waste liquor can be made biologically harmless so that it can be discharged straight into streams by treating it first with CO_2 or flue gas to neutralise it and then with chlorine to oxidise the organic impurities present.

C. O. C.

Cellulose Derivatives as Flocculating Agents. Hercules Powder Co. *BP* 703,964

Addition of a water-soluble or of an alkali-soluble water-insoluble cellulose derivative to water being purified by a coagulation process results in the floc settling quicker and being more difficult to disintegrate and enables reduction of the amount of coagulant needed.

C. O. C.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS**Index of Chemicals in Reports and Documents on the German Chemical Industry. I—Index to Published Reports. II—Index to Unpublished Reports and Documents.**

This index, prepared jointly by the Technical Information and Documents Unit of the Department of Scientific & Industrial Research and by the Association of British Chemical Manufacturers contains references to chemicals

* Microfilms, reports and documents available for inspection at T.I.D.U., D.S.I.R., Cunard Building, 15 Regent St., London S.W.1. Copies of the 26-mm. microfilms can also be bought from T.I.D.U. at a cost for Part I of £10 15s 0d. and for Part II of £25 10s 0d.

reported upon in the BIOS, CIOS, FIAT, etc. series of reports on the German chemical industry (of which there were approx. 1000) and in the unpublished documents held by T.I.D.U. In the main, the chemicals are arranged alphabetically by constitution using the Chemical Society's nomenclature. In many cases, it has been necessary to use German trade names. In other cases products are grouped together, e.g. adhesives, chemical plant, dyes, synthetic fibres.

All plastic materials, e.g. Bakelite, Albertols, Plastopals, Luvithers, etc., have been grouped together under the heading of "Resins, synthetic", but their actual method of manufacture is in some cases described under the name of the monomer, e.g. Styrene and Vinyl Chloride.

Part I of the index consists of over 31,000 cards, most of which bear multiple references. It is contained in two microfilm reels, totalling 1,718 frames. Part II consists of over 72,000 cards, most bearing multiple references. It is contained in five microfilm reels, totalling 4,512 frames.

C. O. C.

Glyoxal and its Derivatives in the Textile Industry.

L. Marcheguet. *Teintex*, 18, 787-793 (Dec. 1953).

After describing the chemistry of glyoxal and its derivatives, various uses of these compounds in the textile industry are described, including control of dimensional stability of regenerated celluloses, which is the basis of the Sanforset (Cluett-Peabody) process, reduction of shrinkage of wool, and assistance in spinning regenerated protein fibres such as Lanital and Ardin.

B. K.

Effect of Solubilisation on the Size and Interaction of Polysoap Molecules.

U. P. Strauss. 5th Canadian High Polymer Forum. *Chem. and Ind.*, 11-12 (2 Jan. 1954).

The effect of solubilisation on the viscosity (η) of polysoap (I) soln. depends strongly on the nature of the solubilised substance. Aliphatic hydrocarbons depress the reduced η until saturation is reached, the depression being independent of the concn. of (I), and dependent only upon the hydrocarbon-(I) weight ratio; it is ascribed to contraction of the mol. of (I). With aromatic hydrocarbons η goes first through a max. before being depressed below its original value, and the height of the max. increases sharply with increasing concn. of (I), disappearing altogether at very low concn. This is attributed to interaction between mol. of (I). With polar-non-polar solubilities η goes first through a max. and then through a min. before the final value is reached, which may be higher or lower than the original η . The max. is due to interactions, but the min. to changes in the size of the mol. of (I). The dependence of the various types of η effects on temp. and the result of electrical conductivity studies confirm these interpretations.

J. W. D.

Potatoes as a Crop and an Industrial Raw Material.

J. A. Radley. *Chem. and Ind.*, 64-68 (16 Jan. 1954).

An account of the manufacture of ethanol, *n*-butanol, acetone, 2:3-butylen glycol (and thence to butadiene), potato starch, dextrin, starch ethers, esters, and xanthanes. The application of the products is indicated. J. W. D.

Chemistry of Chelation in Textile Processing.

A. J. Gard. *Amer. Dyestuff Rep.*, 42, P867-P870 (21 Dec. 1953).

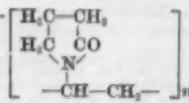
The importance of chelation in textile auxiliaries is discussed with special reference to ethylenediaminetetra-acetates. The heavy-metal-sequestering action of this compound is described, a comparison with phosphates is made, and applications to dyeing and bleaching are discussed. It is emphasised that speed of chelation and stability of the complexing agent are much more important than calcium repression values.

J. W. B.

Polyvinylpyrrolidone—A Versatile Compound with Potential Textile Uses.

E. C. Hansen, C. A. Bergman, and D. B. Witwer. *Amer. Dyestuff Rep.*, 43, P72-P75 (1 Feb. 1954).

Polyvinylpyrrolidone (P.V.P.)—



has been commercialised rapidly during the last three years. The synthesis from acetylene is outlined, and the properties, both textile and non-textile, are discussed. It has high solubilising and complexing powers, and is very effective in stripping vat, sulphur, direct, and developed dyeings on cotton. The complexing action is too strong for P.V.P. to be used as a levelling agent, as no simple way is known of breaking the dye-P.V.P. complex and thus making the colour available for the fibre. The action of P.V.P. is inhibited in colloidal thickenings, and it is thus of no value in printing pastes. It is valuable in rag stripping and is potentially useful in altering the dyeing, physical, and chemical properties of acrylic fibres. J. W. B.

Physicochemical Properties of the Surface of Aqueous Solutions. VI—Breaking of the Solid Surface.

H. Kimizuka. *Bull. Chem. Soc. Japan*, 26, 449-453 (Nov. 1953).

The variation of the frequency of breaking of the surface film of 1% saponin soln. when held under constant tension in a du Noüy tensiometer, and also variations in breaking time as a function of stress, indicate that irreproducible surface tension data are due to the breaking of a solid surface film. A new method of measuring the surface tension of solutions having a solid adsorbed layer is described.

A. J.

Sequestration by Sugar Acids.

C. L. Mehltretter, B. H. Alexander, and C. E. Rist. *Ind. Eng. Chem.*, 45, 2782-2784 (Dec. 1953).

Sugar acids contain contiguous hydroxyl and carboxyl groups, which have the property of chelating metal ions. In general, the sugar acids examined showed maximum sequestration in 2-5% NaOH soln. but were ineffective in neutral or acid soln. Saccharic acid, in particular, was an efficient sequestrant for Ca and Fe ions. Methods are described for determining sequestering capacity for Ca, Fe, and Cu ions in acid, neutral, and alkaline soln.

W. K. R.

Physicochemical Properties of Surface-active Agents.

A. M. Mankowich. *Ind. Eng. Chem.*, 45, 2759-2766 (Dec. 1953).

Data are presented on some physicochemical properties of solutions of 23 detergents of 13 chemical types, alone and in the presence of neutral and alkaline builders and carboxymethyl cellulose, and at various pH levels. The properties measured were—surface tension, interfacial tension, spreading coefficient, contact angle, adhesion tension, and work of adhesion.

W. K. R.

Substituted Silyl Derivatives of Starch.

R. W. Kerr and K. C. Hobbs. *Ind. Eng. Chem.*, 45, 2542-2544 (Nov. 1953).

Methods are described for treating starch with substituted silanes. Three types of derivative were obtained—(i) monofunctional substituted silanes, e.g. trimethylchlorosilane, gave polymers which were water-soluble or dispersible at low levels of substitution and soluble in hydrocarbons at high levels of substitution; (ii) substituted silanes which were essentially monofunctional, but contained a potentially functional group, e.g. diphenyl-n-butoxychlorosilane, gave a "solvent-soluble" product, which with heating and time became an insoluble resin; (iii) substituted silanes having at least two highly reactive groups, e.g. diphenyldichlorosilane, formed insoluble cross-linked polymers. The stability of the siloxy linkage to starch appeared to increase with the size and to vary with the configuration of the other substituent groups attached to the silicon.

W. K. R.

Mothproofing Agents for Wool. XIII—Relation between the Chemical Constitution of Mothproofing Agents and their Effects on Wool.

K. Matsui. *J. Soc. Org. Synthetic Chem. (Japan)*, 10, 333-335 (1952).

PATENTS

Surface-active Wool-scouring Agents.

Monsanto. *BP* 702,930

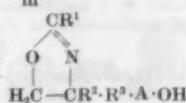
Grease and soil are removed from raw wool by treating first with an aqueous alkali, e.g. soda ash, and then with an aqueous soln. containing an alkali and the product obtained by condensing 1 mol. of *tert*-dodecylmercaptan with 7-9 mol. of ethylene oxide.

J. W. B.

Surface-active Agents. Atlas Powder Co.

USP 2,636,038

Compounds of formula—



(R^1 = aliphatic hydrocarbon of 7-17 C; R^2 = H or CH_3 ; R^3 = short-chain alkylene; A = polyhydroxyethylene or polyhydroxypropylene containing 2-20 hydroxyalkylene groups) are detergents, wetting and emulsifying agents, and corrosion inhibitors.

C. O. C.

Detergent. Colgate-Palmolive-Peet Co. BP 704,390

Addition of a fatty acid amide having 10-20 C in the amide group and of an inorganic polysulphate to a sulphated or sulphonated alkyl aromatic or fatty acyl-containing detergent improves its surface-active properties.

C. O. C.

Anionic Synthetic Detergents. Colgate-Palmolive-Peet Co.

BP 704,021

A higher alkylurea, e.g. dodecylurea, is added to an anionic sulphated or sulphonated synthetic water-soluble detergent composition to enhance the surface-active properties such as detergency and foaming. W. G. C.

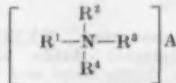
Synthetic Detergents for Wool. 8. BP 704,288

A detergent composition especially effective towards dirty wool comprises a mixture of a water-soluble salt of an alkylbenzenesulphonic acid of 9-18 C and a water-soluble salt of sulphuric ester of a monoalkyl ether of an alkylene glycol (Alk of 10-22 C and alkylene of 2-4 C), e.g. lauryldiglycol ether-sodium sulphate. W. G. C.

Scourable Textile Lubricants. Atlas Powder Co.

BP 702,929

Fibres, yarns and fabrics are rendered more amenable to processing, i.e. from the point of view of softness, scourability, wettability, and antistatic, by treating with a mixture of a scourable lubricant and a cationic surface agent. The lubricant must consist of an ester of a polyhydroxy compound containing 2-6 C atoms with a fatty acid containing 12-30 C atoms or a polyoxalkylene ether thereof, e.g. diethylene glycol monolaurate, which is removable by hot water. The cationic agent has the formula—



(A = anion; R^1 = aliphatic radical of 12-18 C; R^2 and R^3 = separately short chain Alk or alkylol, or R^2 and R^3 form with the N atom a heterocyclic ring; R^4 = short chain Alk or lower aralkyl), e.g. stearylbishydroxyethyl benzylammonium chloride. J. W. B.

Detergent Composition. Colgate-Palmolive-Peet Co.

USP 704,391

Addition of a small amount of higher aliphatic monohydric or dihydric alcohols of > 7 C and of fatty acid amides containing 10-20 atoms in the acyl group to sulphated or sulphonated alkyl aromatic or fatty acyl-containing detergents imparts improved foaming and detergent properties.

C. O. C.

Detergent. Lever Brothers & Unilever. BP 704,257

A combination of alkali metal pyrophosphates and/or tripolyphosphates (40-65% by wt.) and a mixture of anionic detergents (10-20) the remainder being supplemental builders and water displays a synergistic effect in detergency and lathering so that better balance between lathering and cleansing power is obtained, even in soft water, as with most known soapless detergents. The mixture of anionic detergents consists of an alkyl aryl sulphate (50-85% by wt.) and a compound of formula $\text{Alk—CO—NR—(CH}_2\text{)}_n\text{SO}_3\text{A}$ ($\text{R} = \text{H}$, Alk, Ar, or cycloaliphatic; $n = 1-5$; A = H or Alkali metal) (50-15%). C. O. C.

Dispersing and Emulsifying Agents. Monsanto.

BP 705,117

A mixture of an amine salt of an alkylated aromatic sulphonic acid and an alkylene oxide condensate of a

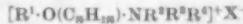
long chain aliphatic compound can be used as an emulsifying agent to yield concentrated emulsions or self emulsifiable powders which will be stable to long storage.

C. O. C.

Quaternary Ammonium Compounds—Bactericidal Detergents. Ward Blenkinsop & Co.

BP 703,477

Compounds of formula—



($\text{R}^1 = p$ -tert.-octylphenyl; $n = 2-4$; R^2 and R^3 = same or different, Alk of < 5 C; R^4 = Alk of < 4 C or aralkyl; X = anion) have high bactericidal and detergent activity. They are useful for sterilising textiles.

C. O. C.

Anti-static Composition for Cellulosic Films. General Aniline.

BP 705,159

A cellulose acetate solution suitable for treating one side of a film of a cellulose derivative to improve its anti-static and slippage characteristics is prepared by mixing a dope solution of cellulose acetate containing a solvent for the said film, and a dispersion of poly-(methylene salicylic acid) and calcium silicate in an aralkyl alcohol of the benzene series.

S. V. 8.

Mordants for Basic Dyes. DuP. USP 2,635,535

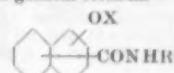
Naphthol sulphonamides of formula—



(X = H, RCO (R = Alk of 1-4 C) or carbalkoxy of 1-5 C; A = divalent radical of an organic diamine of > 7 C and contains at least one benzene nucleus; a position *o* or *p* to the OX groups in the naphthalene nuclei is reactive) have strong affinity for fibres and water-permeable colloids and act as mordants for basic dyes.

USP 2,635,536

Compounds of general formula—



(X = H or RCO (R = Alk of 1-4 C) or carbalkoxy of 2-5 C; R¹ = radical of an aromatic amine) have similar properties.

C. O. C.

Aminoplast Condensates for Crease-resisting Finishes, Coatings, Impregnating Paper, etc.

BP 703,083

Use of aldehydes containing one or more methylol groups to form amino condensates has the advantages that the primary condensates are more soluble in water, the final condensates are more elastic than similar formaldehyde condensates and there is scarcely any formaldehyde liberated. Used on textiles they impart remarkable crease resistance, paper treated with them is very pliable and coatings and films made from them have high elasticity.

C. O. C.

Water-repellent Compositions. British Thomson-Houston Co.

BP 705,277

An excellent water repellent effect is obtained, especially on textiles, by use of a mixture of (a) a urea- or melamine-aldehyde condensate, (b) a liquid organopolysiloxane, and (c) a liquid hydrolysis product of a methylidihalogenosilane of the type described in BP 585,947 (J.S.D.C., 64, 52 (1948)), the finish being superior to that obtained by use of any one of the ingredients alone. The siloxane used should have attached to the Si atoms only monovalent hydrocarbon radicals by means of a C-Si linkage and contain an average ratio of 1.75-3.0 hydrocarbon radicals per Si atom, e.g. those described in BP 585,947.

C. O. C.

Water-repellent Composition. H. G. Figidor.

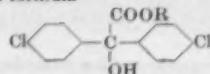
USP 2,635,055

Addition of a small amount of a synthetic non-ionic, non-ionic emulsifying agent to a wax, a water-soluble polyvalent metal salt and an anionic surface-active agent forms an emulsion stable to prolonged storage and dilution. As no stabilising agent or protective colloid is needed the emulsion does not tend to putrefy.

C. O. C.

Insecticides. Gy.

Compounds of formula—



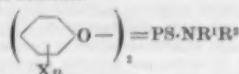
(R = Alk, alkenyl, halogenoalkyl, alkoxyalkyl, cycloalkyl or cycloalkenyl) are strongly insecticidal and are particularly efficacious against moths. They are suitable for application to textiles, paper, furs, leather, etc. from organic solvent solution or aqueous suspension.

C. O. C.

oo-Di(polyhalogenophenyl) N-substituted Amidothiophosphates—Protection against the Attacks of Black Carpet Beetles. Dow Chemical Co.

BP 705,646

Compounds of formula—



(X = Br or Cl; n = 3, 4 or 5; R' = H, Alk or cycloalkyl; R'' = cycloalkyl or Alk) give protection against the attacks of black carpet beetles.

C. O. C.

Fungicidal Compositions. Scientific Oil Compounding Co.

BP 703,463

The product obtained by interacting a water-insoluble metal quinolinate, a water-insoluble metal carboxylic acid soap, and a phenol, can be applied either from aqueous emulsion or solution in an organic solvent to render textiles, paper, leather, etc. fungicidal and/or fungistatic.

C. O. C.

Thermoplastic Compositions of Water-Soluble Cellulose Ethers. Dow Chemical Co.

BP 703,962

A water-soluble cellulose ether composition capable of being thermally fabricated comprises (a) methyl cellulose or methyl hydroxy ethyl, methylhydroxypropyl, hydroxyethyl, or hydroxypropyl cellulose (43–64% by wt.), (b) propylene glycol (2·6–4·5) and (c) glycerine (6–8) A triangular composition diagram is appended.

J. W. B.

Detergent and Bactericidal Compositions. ICI.

BP 703,256

Linear polymers in which the recurring unit has the formula —X-NH-CNH-CNH-Y-NH-CNH-CNH-NH (X and Y = bridging groups in which the total number of C atoms directly interposed between the adjacent N atoms is 10–16 inclusive) mixed with a cationic or non-ionic surface-active substance, e.g. polymeric hexamethylene biguanide hydrochloride mixed with dodecylpyridinium bromide, are very effective detergents and bactericides.

C. O. C.

Driers. Ferro Enamel Corp.

BP 704,333

Salts of organo-substituted phosphorus acids, e.g. dioctyl acid pyrophosphate and mono-octyl-, monoamyl or monobutyl acid orthophosphate, inhibit the decomposition of driers caused by presence of small amounts of water and also decrease the viscosity of the solution of the drier.

C. O. C.

Alphabetical List of New Dyes and Auxiliary Products developed since November 1952 (IV below).**Gas-fading Inhibitors (VIII p. 209).****Water-resistant Leather by Use of Aminosilane Resins (XII p. 214).****IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS****Alphabetical List of New Dyes and Auxiliary Products developed since November 1952. Amer. Dyestuff Rep., 42, 809–842 (7 Dec. 1953).****Mechanical Model for the Evaluation of the Absorption Maxima of Organic Colouring Matters on the basis of their Chemical Constitution.** H. Labhart. *Helv. Chim. Acta*, 36, 1689–1698 (Dec. 1953).

A mechanical model is described whose equation of motion is of the same type as the one-dimensional Schrödinger equation. This model can serve as a

calculating machine for the determination of the eigenfunctions and energy values of the π -electrons in dye molecules, whose total number can be treated according to the theory of H. Kuhn as a one-dimensional degenerate electron gas. Some applications to dye molecules are included.

H. H. H.

Rearrangement of Hydrogens in Phenols. I. I. Kukhtenko. *Doklady Akad. Nauk S.S.R.*, 93, 487–489 (21 Nov. 1953).

Heavy phenol $\text{C}_6\text{H}_5\text{OD}$, on being heated at 210–250°C., is converted into an equilibrium mixture (e.g. in 9 hr. at 250°C.) containing phenol molecules having deuterium attached to the nucleus. Hydroxyl-D is removed from the isomerates by dissolving it in alkali and then reprecipitating with acid, and the deuterium content of the water obtained by combustion of the product is determined. The figures obtained correspond closely to those required for distribution of D between O-H and C-H in a ratio of 0·8, which is close to values found by various workers, and for equal distribution between three positions (*para*, and two *ortho*) in the nucleus. Exchange with m-H occurs to a negligible extent, for the deuterium content of the 2:4:6-tribromophenol obtained by brominating the isomerates (freed from hydroxyl-D) is very low. The equivalence in the exchange of *o*- and *p*-positions is confirmed by an examination of the *p*-bromophenol obtained by monobromination. Similar experiments with *o*-cresol show that this behaves in an analogous manner.

A. E. S.

Reaction of Sulphonation. XXXII—Isomerisation of Naphthalenedisulphonic Acids. A. A. Spryakov and B. I. Karavaev. *J. Gen. Chem. U.S.S.R.*, 23, 1182–1188 (July 1953).

Naphthalene-1:5-, -1:6-, -1:7-, -2:6-, or -2:7-disulphonic acid (1 mole) is heated in 77–93·5% H_2SO_4 (3–7 moles) at 100, 130, 140, or 160°C., and the changes occurring are followed by analysis. The kinetics are complex, but for all acids the same equilibrium mixture appears to be approached. In 1200 hr. at 100°C. in 93·5% H_2SO_4 (7 moles) the 1:6 acid yields a mixture of 9·4% 2:6, 77·3% 2:7, 2·3% 1:6, and 11·0% 1:3 and 1:7 acids. At 160°C. in 77% H_2SO_4 (3 moles) the equilibrium mixture obtained in all cases contains 31% 2:6, 49% 2:7, 9% 1:6, and 11% 1:3 and 1:7 acids. The 1:5 acid isomerises very rapidly—forming mainly the 1:6 acid, which then reacts further—and it is not present in appreciable amount in the equilibrium mixtures.

A. E. S.

Reaction of Sulphonation. XXXIII—Equilibrium between Sulphonic Acids and their Acid Chlorides. A. A. Spryakov and Yu. L. Kuz'mina. *J. Gen. Chem. U.S.S.R.*, 23, 1536–1539 (Sept. 1953).

Equilibrium constants for the reaction—

$$\text{R}(\text{SO}_3\text{Cl})_n + \text{H}_2\text{SO}_4 = \text{R}(\text{SO}_3\text{Cl})_{n-1}(\text{SO}_3\text{H}) + \text{HO-SO}_3\text{Cl}$$

(R is a uni-, bi-, or ter-valent aromatic radical) have the values—for *m*-nitrobenzenesulphonyl chloride, 0·51; 2:5-dichlorobenzenesulphonyl chloride, 0·67; *p*-toluenesulphonyl chloride, 1·18; *m*-chlorosulphonylbenzoic acid 1·22; toluene-2:4-disulphonyl dichloride, 0·90; *m*-benzenesulphonyl dichloride, 1·33; naphthalene-1:3:5-trisulphonyl trichloride, 1·90.

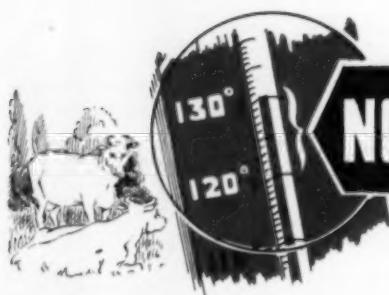
A. E. S.

Mechanism of the Rearrangement of Aromatic Amino and Hydroxy Sulphonates. E. A. Shilov, M. N. Bogdanov, and A. E. Shilov. *Doklady Akad. Nauk S.S.R.*, 92, 93–96 (1 Sept. 1953).

An amino or hydroxy sulphonate acid (naphthionic, 1-naphthylsulphonic, sulphanilic, 2-naphthylamine-1-sulphonic, or 1-naphthol-4-sulphonic acid—free or as Na salt) is heated with Na_2SO_4 labelled with ^{35}S and e.g. naphthalene, 1-naphthylamine, 1-naphthylamine + Na_2CO_3 , or 1-naphthol (which yields 1-naphthol-2-sulphonic acid in all cases), and the products formed are identified and examined for radioactivity. It is shown that even under the heterogeneous conditions of the experiments sulphate-ion exchange between sulphates occurs very readily; new sulphonate acids formed by hydrolysis of the original acid and resulphonation will therefore be radioactive. Such products are obtained under acid conditions; under neutral conditions, however, good yields of products of low radioactivity are often obtained, whereas under alkaline conditions reaction is generally inhibited. The case of naphthionic acid may be taken as

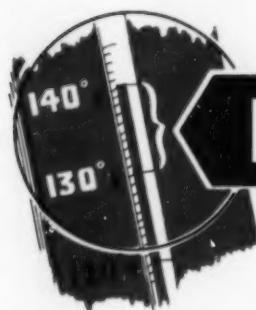
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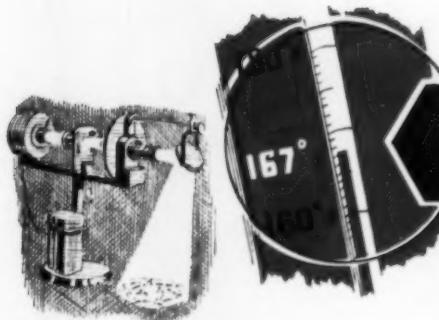
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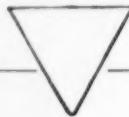
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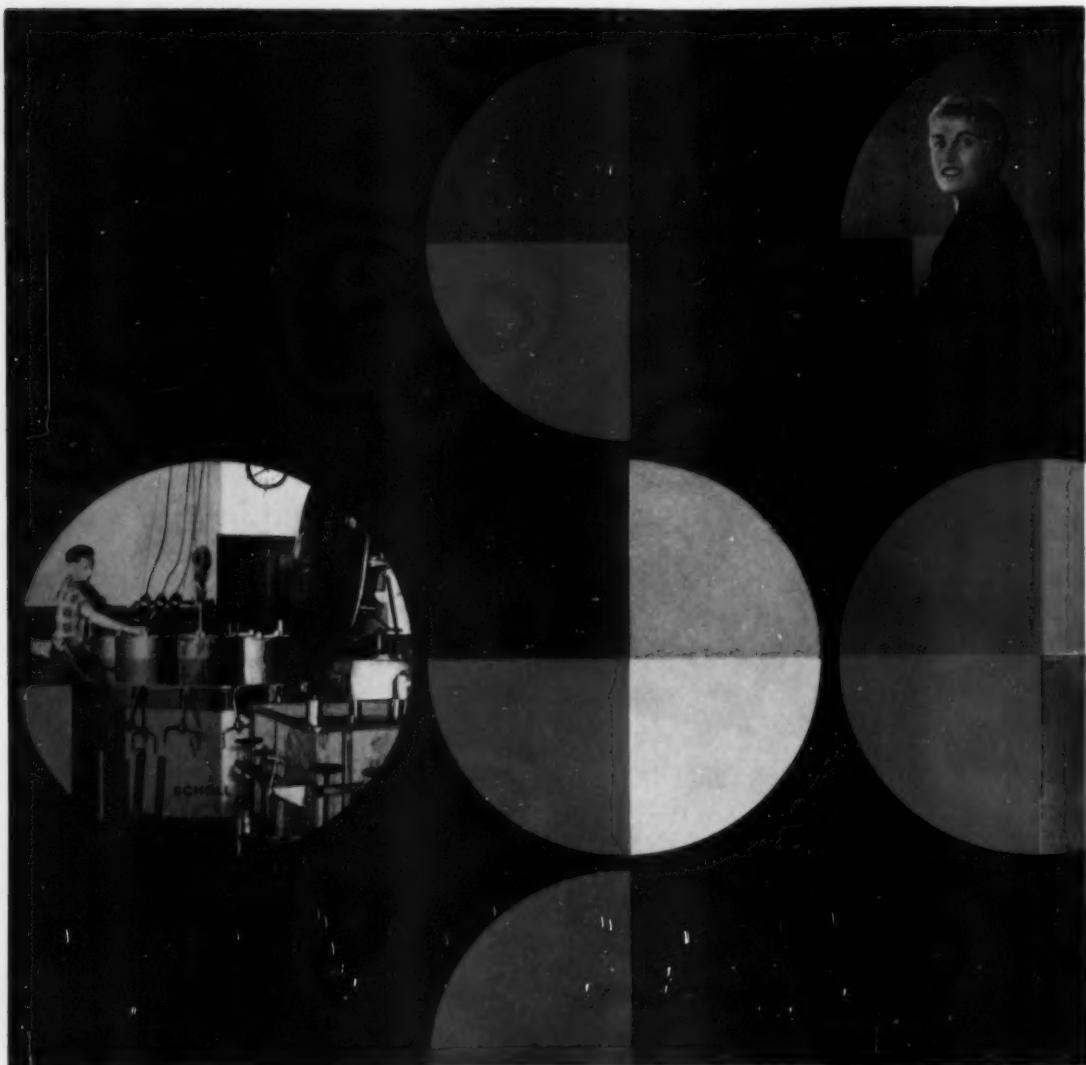
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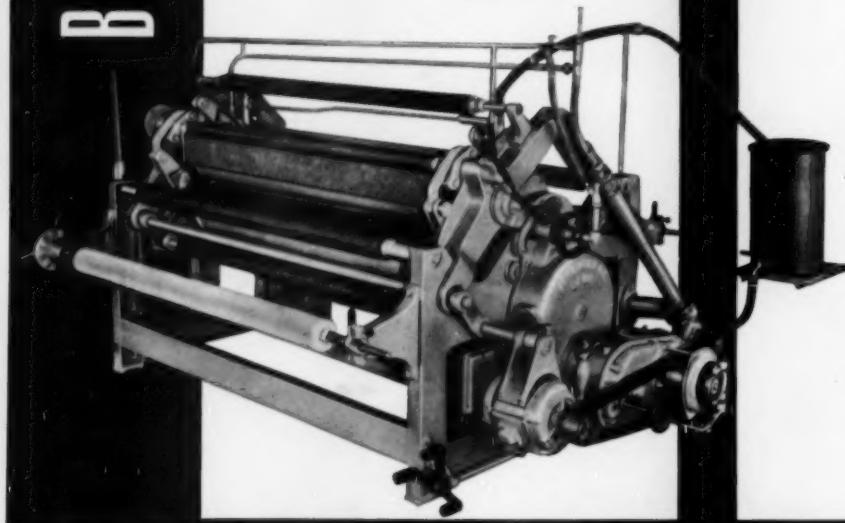
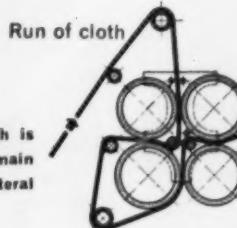
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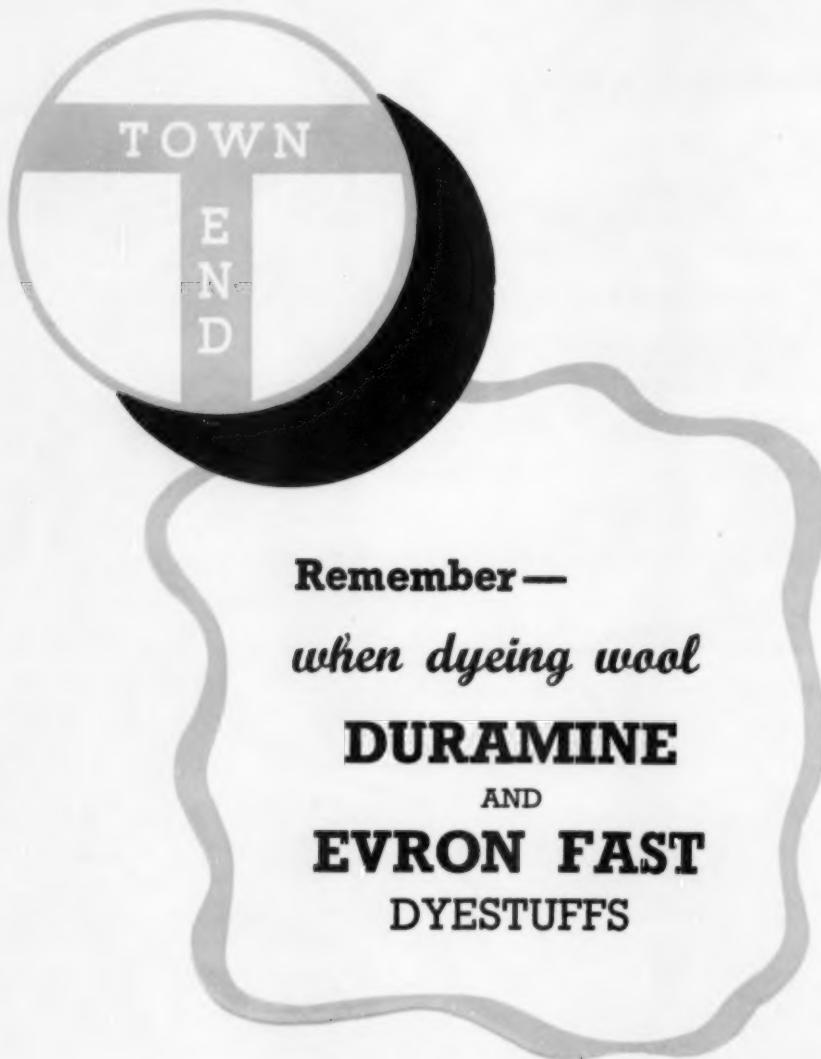
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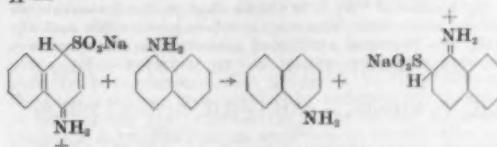
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example. Under acid conditions (free acid + 1-naphthylamine) the product is 1-naphthylamine-2-sulphonic acid of high activity (on being ashed, etc. it yields Na_2SO_4 having 74% of the activity of the Na_2SO_4 component of the original mixture), and hydrolysis leading to intermediate formation of H_2SO_4 evidently occurs. Under neutral conditions (Na salt + 1-naphthylamine) 1-naphthylamine-2-sulphonic acid of low activity is obtained in high yield (although reaction is inhibited by addition of a little Na_2CO_3). Intermediate hydrolysis evidently does not occur under these conditions, and it is suggested that reaction occurs with a quinonoid ion formed by addition of H^+ .



In absence of 1-naphthylamine, a similar reaction may occur, less readily, between two molecules of naphthionate, yielding a disulphonate and 1-naphthylamine, which can then participate in reaction in accordance with the above scheme.

A. E. S.

Effect of the Sulpho Group on Aromatic Systems—

Hammett's σ -Values of the SO_3^- -Substituent. H. Zollinger, W. Büchler, and C. Wittwer. *Helv. Chim. Acta*, 36, 1711–1722 (Dec. 1953).

Hammett's σ -values of the *m*- and *p*-sulpho group are determined from acidity constants of the *m*- and *p*-sulphonic acid ions of phenol, benzoic acid, and anilinium ion, measured at different ionic strengths by the potentiometric and the spectrophotometric methods. The values of the *p*-substituent show a mesomeric effect weaker than that of other negative substituents. This is due to the fact that the acidity constants of these benzenesulphonic acid deriv. relate to the ionisation of a second proton of the mol. Therefore the applicability of Hammett's equation is limited in the case of the SO_3^- -substituent. H. H. H.

Reduction of Aromatic Nitro Compounds by Magnesium and Methyl Alcohol. K. F. Keirstead. *Canadian J. Chem.*, 31, 1064–1077 (Nov. 1953).

Reduction of 12 substituted nitrobenzenes by Mg in CH_3OH shows some of them to reduce smoothly to the corresponding azoxy, while others yield a complex mixture of azo, azoxy, and other compounds. Unsuccessful reductions were prominent with *ortho*-substituted compounds, whereas the method appears to be promising for the reduction of nitroanilines where the amino is protected by an acetyl group. A certain similarity appears to exist in the properties of the unknown component found in reduction mixtures, where the azoxy compound is absent or present in small yield; this bright yellow or brown substance is highly sol. in alcohol but unlike a tar is not removable by treatment with activated charcoal or chromatographic alumina. No amines were found in the reduction mixtures. 17 references. H. H. H.

Reduction of *m*-Nitrobenzaldehyde. N. G. Gaylord and J. A. Snyder. *Rec. Trav. chim.*, 72, 1007–1008 (Nov. 1953).

The reduction of *m*-nitrobenzaldehyde with lithium aluminium hydride gives *mm'*-azobenzyl alcohol (28.5% yield) rather than the reported *m*-aminobenzyl alcohol. H. H. H.

Dye Intermediates from 1- and 2-Naphthols. R. M. Desai. *Bombay Technologist*, 1, 82–83 (1950); *Chem. Abs.*, 48, 1686 (10 Feb. 1954).

4-Acyl and 4-acyl-1-naphthols and 6-acyl-2-naphthols were prepared by treating naphthol or their methyl ethers at 0°C. with acyl or aryl chlorides in presence of ZnCl_2 or AlCl_3 as condensing agents in nitrobenzene soln. 6-Acyl-2-naphthols could not be obtained by this method, 1-aryl-2-naphthols always being produced irrespective of the solvent used. 2-Acyl-1-naphthols were prepared by the Nenki reaction from the naphthols and the appropriate

acids. The sulphonamido derivatives were prepared from Schaeffer's acid through its sulphonyl chloride by reaction with amines. The arylnaphthols were used as secondary coupling components in azoic dyeing, and the fastness to washing, bleaching, rubbing and light measured.

C. O. C.

The Coupling Reaction. VI—Primary Salt Effects as Means for the Kinetic Determination of the Reacting Form of the Diazo Components. H. Zollinger. *Helv. Chim. Acta*, 36, 1723–1730 (Dec. 1953).

The rates of the azo coupling reactions of diazo-*p*-toluene (I), diazobenzene-4-sulphonic acid (II), and diazobenzene-2:5-disulphonic acid (III) with 2:6-naphthylaminesulphonic acid have been measured at ionic strengths between 0.005 and 0.25, and the data show that reaction I has a negative and II a positive primary salt effect respectively, corresponding fairly well to Brønsted's equation at ionic strengths below 0.02; reaction III is practically independent of the ionic strength. This is the first direct proof for the intervention of the diazonium ion as the active reactant in coupling in aq. soln. It appears that the coupling mechanism in this medium is not the same as that in organic solvents (cf. Huisgen, *Annalen*, 574, 184 (1951)).

H. H. H.

The Coupling Reaction. VII—Kinetic Measurement of the Influence of Substituents on the Reactivity of the Benzenediazonium Ion. H. Zollinger. *Helv. Chim. Acta*, 36, 1730–1736 (Dec. 1953).

The coupling rate constants of 10 benzenediazonium ions substituted respectively by *m*- and *p*- OCH_3 , $\cdot\text{CH}_3$, $\cdot\text{Cl}$, and $\cdot\text{NO}_2$, $\cdot\text{SO}_3^-$, and 2:5-di- SO_3^- , and of the unsubstituted ion, with 2:6-naphthylaminesulphonic acid and with 2:6-naphtholsulphonic acid under the same conditions (ionic strength 0.25; 20°C.), agree with Hammett's equation for substituted benzene deriv. in contrast to the reactions with other coupling components.

H. H. H.

Azo Dyes. II—Azo Dyes from Naphthidine. K. Murato and K. Harada. *Bull. Fac. Engr., Hiroshima Univ.*, (1), 117–119 (1952); *Chem. Abs.*, 48, 1686 (10 Feb. 1954).

A number of symmetrical diazo dyes obtained by coupling tetrazotised naphthidine with 2 mol. of various naphthol- and aminonaphthol-sulphonic acids were examined for hue and affinity for wool and cotton. Thus, using 2-naphthol-6-sulphonic acid, a reddish purple for wool was obtained, and H acid (alkali-coupled) gave a blue direct cotton dye.

E. S.

Azo Dyes. III—Dyes from 2:6-Dichloro-*p*-phenylene-diamine. *Ibid.*, (2), 1–5 (1953); *Chem. Abs.*, 48, 1687 (10 Feb. 1954).

A number of 4-amino-3:5-dichlorophenylazo derivatives of various naphthol-, naphthylamine-, and aminonaphthol-sulphonic acids were examined for hue and affinity for wool and cotton. Thus 2-naphthol-6-sulphonic acid gave a brown for wool, and H acid (alkali-coupled) gave a blue direct cotton dye.

E. S.

Azoic Dyes. I—Coupling Components derived from Amino- and Diamino-diphenyls. T. Maki and K. Obayashi. *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 55, 401–403 (1952); *Chem. Abs.*, 48, 1005 (25 Jan. 1954).

Arylamides of 3-hydroxy-2-naphthoic acid were prepared by condensation with 4-aminodiphenyl (I), 2-aminodiphenyl (II), 4:4'-diaminodiphenyl (III), and 2:2'-diaminodiphenyl (IV) using 2 mol. of 3-hydroxy-2-naphthoic acid in the case of the diamines. The products were examined as azoic coupling components. The "straight chain" naphthols from I and II gave stable impregnating baths only by the cold method; they have high substantivity. With various diazo compounds, the naphthol derived from III gave deep browns and blacks of excellent light fastness (6–8). The "branched chain" naphthols from II and IV were easily soluble in aq. NaOH and gave hues similar to those given by Naphthol AS, but less fast to light.

E. S.

Azo Derivatives of 1-Phenyl-3-furyl-5-pyrazolone-disulphonic Acids. D. D. M. Casoni. *Ricerca sci.*, 22, 1785-6 (1952); *Chem. Abs.*, 48, 1341 (10 Feb. 1954). A series of pyrazolone azo dyes of formula—



(R = CH_3CO , C_6H_5CO or $C_6H_5O_2Ar$ = various aryl groups) was prepared by coupling 1-p-sulphophenyl-3-furyl-2-pyrazolin-5-one with an appropriate aryl diazonium salt at 2-3°C. Presence of two SO_2H groups increases the solubility and dyeing properties. C. O. C.

Azo-coupling of 2:2-Bis-p-hydroxyphenylpropane. I. V. Khvostov. *Doklady Akad. Nauk S.S.R.*, 93, 679-680 (1 Dec. 1953).

Bis-p-hydroxyphenylpropane (condensation product of phenol and acetone) couples with diazo cpd. with elimination of acetone and formation of the azo cpd. normally obtained from phenol itself (cf. behaviour of analogous condensation products formed by 2-naphthol and 2-naphthol-3-carboxy anilides with formaldehyde). Treatment of the cpd. with nitrous acid yields p-nitrosophenol. A. E. S.

Coupling of Diazo Compounds with Substances having Active Methyl Groups. A. A. Kharhakov. *J. Gen. Chem. U.S.S.R.*, 23, 1175-1181 (July 1953).

Previous work of the A. E. Porai-Koshits school has shown that active methyl groups (i.e. those which condense with e.g. aldehydes to give $\alpha\beta$ -unsaturated cpd.) will, if sufficiently active, react with diazo compounds, forming tautomeric dyes of the type—



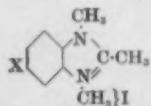
Favourable conditions for coupling are found when the methyl is in the α - or γ -position with respect to N in the heterocyclic ring of e.g. pyridine, quinoline, acridine, and benzo- and dibenzo-acridines; and the methyl of 2:4:6-trinitrotoluene will also react with very active diazo compounds. The relative reactivities of these compounds are discussed from the point of view of electronic theory, which accounts for the fact that some heterocyclic compounds will couple only after conversion into quaternary salts. It is now shown that the ethiodide of non-coupling 2-methylbenzothiazole will couple with diazotised p-nitroaniline, and absorption spectra are given for the resulting dye and for most of the following dyes, the preparations of which are described—

2:5-Dimethoxy-4-aminodiphenylamine	→ 9-Methylacridine
Aniline	→ 2-Acetamido-9-methylacridine
p-Nitroaniline	→ 2-Acetamido-9-methylacridine
p-Nitroaniline	→ 2:4:6-Trinitrotoluene
p-Nitroaniline	→ 2-Methylglyoxaline
p-Nitroaniline	→ 2-Methylpyridine
p-Nitroaniline	→ 2:6-Dimethylpyridine-3:5-dicarboxylic acid

A. E. S.

Reactivity of some Derivatives of 2-Methylbenzimidazole. I—Reaction involving the Hydrogens of the Methyl Group. B. A. Porai-Koshits and Kh. L. Muravich. *J. Gen. Chem. U.S.S.R.*, 23, 1583-1593 (Sept. 1953).

The reactivity of the methyl group in 2-methylbenzimidazole is enhanced (see previous abstract) by conversion of the cpd. into a quaternary salt. 1:2:3-Trimethylbenzimidazolium iodide—

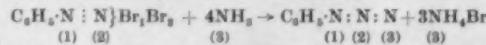
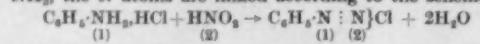


(X = H) is prepared and shown to react very readily with aldehydes (→ dimethin dye) and nitroso cpd. (→ azamethin dye). Salt formation, however, blocks only one pair of unshared electrons, and the presence of the second pair is considered to account for the fact that the cpd. will not couple at the methyl group with a diazo cpd. Introduction of an electron-attracting group (X = NO_2) in order to restrain these electrons enables azo coupling to proceed readily (simultaneous loss of CH_3I occurs).

Replacement of 1-methyl by phenyl in the quaternary salt (X = H or NO_2) leads, unexpectedly, to decreased activity, and the corresponding tertiary bases do not undergo any of the characteristic reactions, even when two NO_2 groups (2:4) are introduced into the 1-phenyl substituent. The preparation and properties of a number of dimethin, azamethin, and azo dyes of the types indicated are described. A. E. S.

Reactions with ^{15}N . XI—Formation of Phenyl Azide from Benzenediazonium Perbromide and Ammonia. K. Clusius and H. Hürzeler. *Helv. Chim. Acta*, 37, 383-388 (Jan. 1954).

By means of ^{15}N , it is shown that in the formation of phenyl azide from benzenediazonium perbromide and aq. NH_3 , the N atoms are linked according to the scheme—



Only the linear structure is compatible with the analytical results, and this viewpoint is in accord with the earlier reactions prepared for the decomp. of phenyl azide.

H. H. H.

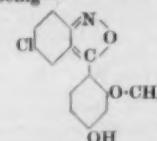
Dissociation of Triarylmethanols. O. F. Ginzburg. *J. Gen. Chem. U.S.S.R.*, 23, 1504-1509 (Sept. 1953).

The basic dissociation constants of the carbinols of Malachite Green (I) and Crystal Violet (II) are determined from the pH values of aq. soln. of pure hydrobromides and hydroiodides of the dyes. The results are in close accord with those of Goldacre and Phillips (see J.S.D.C., 66, 68 (Jan. 1950)). Further data on I and other triphenylmethane dyes in aq. and aq. acetone soln. are obtained from the pH values of soln. containing equimol. amounts of carbinol and salt—increased dissociation occurs in presence of acetone, the pK of e.g. I being 7.07 in water and 6.05 in 60% aq. acetone. In nitrobenzene soln. the carbinols of I and II behave as strong bases, being dissociated, according to absorptiometric measurements, to the extent of 70% and 90% respectively. A. E. S.

Hydroxyfuchsone Dyes. XVII—Condensation of Isomeric Nitrobenzaldehydes with Guaiacol.

I. S. Ioffe and B. G. Belon'kii. *J. Gen. Chem. U.S.S.R.*, 23, 1525-1529 (Sept. 1953).

Condensation in presence of HCl of *m*(or *p*)-nitrobenzaldehyde with guaiacol followed by oxidation of the leuco cpd. formed yields 4'-hydroxy-3:3'-dimethoxy-3' (or 4')-nitrofuchsone, but condensation of *o*-nitrobenzaldehyde stops at the diphenylmethanol deriv., at which stage the *o*-nitro group enters into a cyclisation reaction in which simultaneous oxidation of HCl occurs, the final product being—



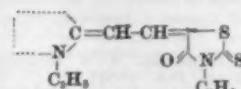
A. E. S.

Synthesis of Cyanine Dyes. IX—Chemical Structure of

Neocyanine. Y. Mizuno and Y. Tanabe. *J. Pharm. Soc. Japan*, 73, 227-231 (1953); **X—A New Procedure for Synthesising Trinuclear Trimethincyanine Dyes.** *Ibid.*, 232-5; **XI—A New Procedure for Trinuclear Trimethincyanine Dyes.** *Ibid.*, 235-7; **XII—A New Method of Synthesis of Unsymmetrical Trinuclear Dyes containing Two Different Heterocyclic Rings.** *Ibid.*, 237-240; **XIII—A New Method of Synthesis of Unsymmetrical Trinuclear Dyes containing Two Different Heterocyclic Rings.** *Ibid.*, 240-2; *Chem. Abs.*, 48, 475-9 (25 Jan. 1954).

meroCyanine Dyes derived from Rhodanine. V—Some Tetra- and Hexa-methin *mero*-Cyanines derived from 3-Ethylrhodanine. M. V. Deichmeister, I. I. Levkoov, and E. B. Lifshits. *J. Gen. Chem. U.S.S.R.*, 23, 1529-1535 (Sept. 1953).

In continuation of a previous investigation (see J.S.D.C., 68, 220 (June 1952), the absorption max. of dimethin *mero*cyanines of the type—

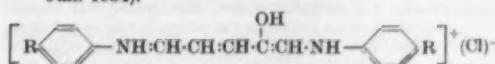


are determined in various solvents. Further analogous tetra- and hexa-methin dyes (second heterocycles derived from quinoline, 3:3-dimethylindolone, or benzothiazole) and the corresponding symmetrical tri- and penta-methin oxanines are prepared, and their spectra are analysed in the way previously described (*loc. cit.*). The same general regularities are observed. It is found also that the hypsochromic shift (compared with the arithmetic mean of the symmetrical-dye values) generally increases as the polymethin chain increases in length, and these changes and the hypsochromic shift produced (in dyes derived from the less basic heterocycles) by reducing the polarity of the solvent are explained in terms of the increased tendency for the zwitterion structure to occur when the medium is polar, the polymethin chain is short, or the second heterocycle is highly basic. A. E. S.

Position of Sensitisation Maxima in Photographic Emulsions sensitised by Polymethin merocyanines. M. V. Deichmeister, I. I. Levkoef, E. B. Lifshits, and S. V. Natanson. *Doklady Akad. Nauk S.S.R.*, **93**, 1057-1059 (21 Dec. 1953).

The tetra- and hexa-methin merocyanines referred to in the previous abstract, and the corresponding dimethin dyes, have sensitisation max. in silver bromide emulsions which are displaced from their absorption max. (in alcoholic soln.) by 32-189 m μ in the direction of the long waves (the usual displacement for cyanine dyes is 25-45 m μ). Also, whereas the bathochromic shifts in the absorption max. due to lengthening of the polymethin chain average 82 and 30 m μ for di- \rightarrow tetra- and tetra- \rightarrow hexa-methin respectively, the corresponding average shifts in the sensitisation max. are each 110 m μ . It is considered that the main factor determining this behaviour is the polarising action of silver bromide on the adsorbed dye, an effect that is greatest in dyes of low polarity. A. E. S.

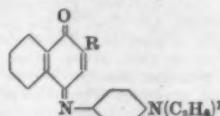
Decomposition of Stenhouse Dyes in Solution. J. C. McIowan and K. Moss. *Chem. and Ind.*, **23-24** (2 Jan. 1954).



derived from aniline (R = H), *p*-toluidine (R = CH₃), *p*-chloroaniline (R = Cl), and *p*-anisidine (R = OCH₃) and their hydrochlorides in oxygen-free alcoholic soln. (~0.001%) sealed in tubes under nitrogen, fade in sunlight and recover their colour in the dark. The fading is accompanied by a drop in pH which is also reversed in the dark, and is not noticeably affected by temp. except in that a decrease in temp. causes a delay in the return of colour (complete in 5 min. as against < 1 min.). The colour may also be restored in sunlight by addition of a little conc. HCl, and dyes dissolved in 0.001 N. alcoholic HCl fade very little in sunlight. Potentiometric determination of the pK_a values in water at 20°C. of the aniline and *p*-chloroaniline derivatives support the theory that the dyes are hydrolysed in soln. Oxygen accelerates the decomp. especially in absence of acid; the products of oxidation have not yet been isolated. J. W. D.

Effect of the Ionisation of Carboxyl and Sulpho Groups in Indoaniline Dyes on their Colour. N. S. Spasokukotskii, I. I. Levkoef, and B. S. Portnaya. *Doklady Akad. Nauk S.S.R.*, **93**, 671-674 (1 Dec. 1953).

The absorption max. of the dyes—



(R = H (absorption max. 625 m μ), COOH, COO-COOCH₃, SO₃⁻, CO-NH-C₂H₅R¹-CO-NH-C₂H₅R²; R¹ = H,

COOH, COO⁻, COOCH₃) are listed. Introduction of R = COOH or CO-NH-C₂H₅ has a powerful bathochromic effect (+ 112 m μ and + 75 m μ , respectively) attributed to hydrogen-bonding with the quinone O. The effect is reduced in the former case by esterification (to + 35 m μ) or ionisation, when it is reversed (- 25 m μ); it is reduced in the latter case by introduction of R¹ = o-COOH or o-COOCH₃ (to + 65 m μ), which are considered to reduce hydrogen-bonding with the quinone O by competing for the amide H (these groups in *m*- and *p*-positions increase the bathochromic effect). In all cases ionisation of COOH leads to a hypsochromic shift. The effect of the introduction of R = SO₃⁻ is very small (- 5 m μ). A. E. S.

Investigations on Colour Development. IX—Behaviour of Sulphite-containing Colour Developers towards Colloid-free Silver Salts in Presence of a Dye-forming Component. W. Brune and U. Kronacher. *Z. wiss. Phot.*, **45**, 145-158 (1953); **X—Behaviour of Sulphite-containing Colour-forming Developers towards Silver Bromide Emulsions in Presence of a Dye-forming Component.** *Ibid.*, 158-162.

Sulphur Dyes. H. de Diesbach, P. Rhynor, and A. Cavagni. *Helv. Chim. Acta*, **36**, 2037-2045 (Dec. 1953).

Differences in constitution between black and blue sulphide dyes obtained from indophenols and similar products are discussed. It is deduced that the black dyes have an angular structure, and that they contain several amino groups and -S- and -SS- linkages. In contrast, the blue dyes have a linear structure with fewer amino groups (one per thiazine ring) and -SO- and -SO-SO- linkages. H. H. H.

Antitubercular Substances. X—Oxidation Products of Derivatives of o-Phenylenediamine. Structure of the Phenazine Pigment B.283. V. C. Barry and J. G. Belton. *Proc. Roy. Irish Acad.*, **55B**, 149-156 (1953); **XI—Phenazine Pigments derived by Oxidation of o-Phenylenediamine Derivatives.** V. C. Barry, J. G. Belton, J. F. C. Chambers, M. L. Conalty, R. Kelly and D. Twomey. *Ibid.*, 157-164; *Chem. Abs.*, **48**, 1377-8 (10 Feb. 1954).

X—Oxidation of 2-aminophenylenediamine yields 2-amido-3:5-dihydro-2-imino-5-phenylphenazine (Pigment B.283). The long disagreement about the results of this oxidation has been cleared up by the discovery that the free base exists in two forms of differing m.p. depending upon the temperature at which they are crystallised but having identical ultraviolet and visible absorption curves.

XI—Data about a number of oxidation products of o-phenylenediamine derivatives are given. C. O. C.

Sulphur Black. C. C. Yin, W. H. Yuan and H. H. Chang. *Chem. Ind. and Eng. (China)*, **2**, (3) 80-5 (1951); *Chem. Abs.*, **48**, 1683 (10 Feb. 1954).

A reddish sulphur black is obtained by hydrolysing dinitrochlorobenzene with NaOH and adding polysulphide. With the polysulphide index fixed at 3.6 and varying the molecular ratio of dinitrochlorobenzene to polysulphide, the effective range for dye formation is between 1:1.59 and 1:1.72; a reddish black is formed only if the ratio is between 1:1.63 and 1:1.64. If this ratio is fixed at 1:1.58 and 1:1.62 the lower limits of polysulphide index for dye formation are 3.9 and 3.1 respectively. In both cases, the intensity of the dye increases with the polysulphide index but the hue is always greenish. Rate of thionation increases with the above ratio but is little affected by change in polysulphide index. The intensity and the redness of the dye are antagonistic. To obtain the reddish hue, the intensity cannot exceed 110%. C. O. C.

Height of Potential Barrier in Hydrogen Bonds of Quinhydrone. I. P. Gragerov and A. I. Brodskii. *J. Gen. Chem. U.S.S.R.*, **23**, 1193-1199 (July 1953).

Mixtures of quinol and heavy benzoquinone (C₆D₆O₂) are heated in silica tubes (glass acts as a catalyst) at various temp. for various periods of time and then fractionated in a high vacuum; the deuterium contents of the quinol and the benzoquinone obtained are determined. The results indicate that transfer of hydrogen in the hydrogen bond of quinhydrone (-O-H \cdots O= \rightarrow =O \cdots H-O-) is a first-order reaction having an activation energy of ~ 63 kg.cal./mole (a potential barrier in the bond of

~ 32 kg.eal.). The experimental value is of the same order as that obtained by the use of available approximation methods of calculation. A. E. S.

Anthraquinoneacridone. I—Vat Dyes from 5-Amino-2:1-anthraquinoneacridone. K. Murata and K. Harada. *Bull. Fac. Engr., Hiroshima Univ.*, (2) 7-14 (1953); *Chem. Abs.*, 48, 1688 (10 Feb. 1954).

The following vat dyes were synthesised by condensing various radicals with the amino group in 5-amino-2:1-anthraquinoneacridone—5-methylamino-R (R = anthraquinone acridone) reddish black, 5-ethylamino-R dark brownish violet, 5-acetamido-R yellowish brown, 5-glycol-R brownish black, 5-anilino-R violet black, 5-p-toluidino-R reddish violet, 5-p-chloroanilino-R dark brown, 5-p-nitroanilino-R dark brownish violet, 5-p-hydroxyanilino-R black, 5-p-carboxyanilino-R brownish violet, 5-anilino-R-p-sulphonic acid brownish violet, 5-p-amino-anilino-R brownish red, 5-p-acetamido-anilino-R black, 5-benzylamino-R grey, 5-benzamido-R brownish black, 5-sulphotoluamido-R black, 5-(1-naphthyl-amino)-R violet black, 5-(2-naphthyl-amino)-R blackish violet, 5-(1-anthraquinonyl-amino)-R violet, 5-(2-anthraquinonyl-amino)-R reddish violet. C. O. C.

Synthesis of Islandicin and other Anthraquinone Colouring Matters. B. S. Joshi. *Bombay Technologist*, 2, 78-9 (1952); *Chem. Abs.*, 48, 1688 (10 Feb. 1954).

The structure of islandicin has been confirmed by unambiguous synthesis to be 1:4:5-trihydroxy-2-methyl-anthraquinone. Bromination of the 2-p-toluene sulphonate of alizarin in acetic acid yielded the 2-p-toluene sulphonate of a monobromo alizarine which, hydrolysed with cone. H_2SO_4 , yielded 4-bromoalizarin. The chloro-, iodo-, anilino-, and toluidino-analogues of 4-bromoalizarin were prepared. Indanthren Turquoise Blue GK and 3 GK were shown to be 11-chloro-6-amino anthraquinone acridone and 10:12-dichloro-6-amino anthraquinone acridone respectively. Derivatives of 6-amino anthraquinone acridone containing Cl in the anthraquinone nucleus, in the benzene half of the molecule and in both of these, were synthesised and the fastness of their dyeings on cotton determined. Dehalogenation of 7-halogeno substituted anthraquinone acridones and degradation of the latter by KOH fusion were also studied. C. O. C.

Absorption and Fluorescence Spectra of Chlorophyll and Magnesium Phthalocyanine in the Adsorbed State. V. F. Gachkovskii. *Doklady Akad. Nauk S.S.R.*, 93, 511-514 (21 Nov. 1953).

An analysis of the absorption and fluorescence spectra of chlorophyll and magnesium phthalocyanine and the effect thereon of complex-formation with oxygen-containing substances; cf. previous work (see *J.S.D.C.*, 67, 36 (Jan.), 199 (May 1951); 68, 318 (Aug. 1952)).

A. E. S.

Active and Inactive Forms of Protochlorophyll, Chlorophyll, and Bacteriochlorophyll in Photosynthesising Organisms. A. A. Krasnovskii, I. M. Koobutskaya, and K. K. Voinovskaya. *Doklady Akad. Nauk S.S.R.*, 92, 1201-1204 (21 Oct. 1953).

Protochlorophyll and bacteriochlorophyll, like chlorophyll (see *J.S.D.C.*, 69, 508 (Dec. 1953)), exist in active (monomeric) and inactive (aggregated) forms. The present state of knowledge concerning the spectral characteristics of these forms and the conditions of their occurrence in various organisms are summarised. A. E. S.

Effect of Aluminium Chloride on Absorption Spectra of Anthocyanins. T. A. Geissman, E. C. Jorgenson, and J. B. Harborne. *Chem. and Ind.*, 1389 (26 Dec. 1953).

Detection or determination of naturally occurring anthocyanins is severely limited by the similarity of their absorption spectra in the 500-550 m μ . region. Addition of $AlCl_3$ soln. to soln. of cyanidin and its glycosides causes a bathochromic shift of the absorption max. by 16-35 m μ . Pelargonidin derivatives are unaffected, indicating that the colour change is due to complex formation of the Al with the free 3':4'-dihydroxyl grouping of the cyanidin derivatives. Results of measurements on several anthocyanins and anthocyanidins are tabulated. Diagrams

show the application of absorption and difference spectra to the analysis of typical pigment mixtures.

J. W. D.

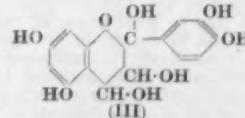
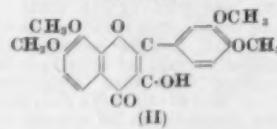
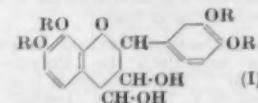
Anthocyanins in Bananas. N. W. Simmonds. *Nature*, 173, 402-403 (27 Feb. 1954).

Pigments are extracted from banana flowers by 1% HCl and examined in the test tube and by paper chromatography. Results are discussed at some length: six anthocyanidins are found, these being pelargonidin, cyanidin, peonidin, delphinidin, petunidin, and malvidin.

J. W. B.

Isolation from *Acacia melanoxylon* of a Flavan-3:4-diol and its Possible Bearing on the Constitution of Phlobatannins. F. E. King and W. Bottomley. *Chem. and Ind.*, 1368 (19 Dec. 1953).

Prolonged extraction of Australian blackwood (*Acacia melanoxylon*) with boiling ether yields an amorphous buff powder consisting principally of melacacidin (I; R = H), the tetramethyl ether of which (I; R = CH_3) yields upon Oppenauer oxidation, 7:8:3':4'-tetramethoxyflavonol (II)—



Despite the close resemblance between flavan-3:4-diols (I) and (III), the possible leuco-anthocyanidin structure proposed by Robinson and Robinson (*Biochem. J.*, 27, 206 (1933)), this is the first recorded instance of a compound of either type being found in Nature. Melacacidin appears to be one of those widely distributed but imperfectly identified plant constituents which function as leuco-anthocyanins, but in their other reactions are indistinguishable from condensed tannins (phlobatannins); this offers an acceptable alternative to the recently discredited flavopinacol hypothesis of condensed tannin structure due originally to Russell (*Chem. Reviews*, 17, 155 (1953)). The tannin reactions of the so-called leuco-anthocyanins may be due to catechins formed during the oxidation of the leuco compounds to anthocyanins by a compensating reduction.

J. W. D.

Polyenes from *Brassica rutabaga*. A. E. Joyce. *Nature*, 173, 311-312 (13 Feb. 1954).

Chromatographic analysis of pigment extract from *Brassica rutabaga* yields several polyenes, and their absorption spectra are discussed. It is suggested that they should be classified in terms of their absorption maxima rather than by their position in the chromatograph adsorption column.

J. W. B.

Pigmentation of the Jellyfish, *Pelagia noctiluca*. N. Millott and D. L. Fox. *Nature*, 173, 169 (23 Jan. 1954).

This jellyfish contains three main pigments—magenta, brown, and blue—the first of which is studied in some detail. Absorption spectra suggest a chromoprotein, and in other ways it behaves like a melanin except that it contains no S. It contains N and a substituted phenol, but no aldehyde or carbohydride groupings. It gives no reactions for proteins, tyrosine, tryptophan, arginine, or other α -amino acids, or purines. The chromogen, prepared by hydrolysis with trypsin, gives, after KOH fusion, a positive Ehrlich reaction for the indole group, but no pyrrole reaction on roasting. The absorption spectrum of the colour developed in the indole reaction is very similar to that given by melanin from black feathers.

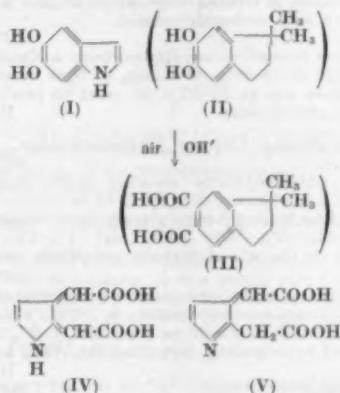
J. W. B.

Melanin Formation. F. K. Duxbury. *Chem. and Ind.*, 1364 (19 Dec. 1953).

Experimental evidence, provided by a study of the effect of H_2O_2 on the oxidation of dihydroxyindole (I) to melanin, suggests that most of the CO_2 evolved during the oxidation arises from the action of H_2O_2 on melanin (and possibly a precursor or melanin), and that the conversion of I to a melanin-like pigment can be achieved without loss of CO_2 . Natural melanins (e.g. sepiin melanin) are similarly attacked by H_2O_2 . J. W. D.

Oxidative Fission of Catechol Compounds and the Structure of Melanins. Z. E. Jolles. *Chem. and Ind.*, 1367-1368 (19 Dec. 1953).

The dihydroxyindole derivative (II) readily oxidises in alk. soln. to the tetracarboxylic acid (III), suggesting that the ring fission of the catechol portion of the indole precursor (I) may proceed via the pyrrole (IV) or the pyrrolenine form (V) in analogy with III.



This and other evidence which is cited is consistent with the view that the formation of natural melanin from tyrosine and congeners is the result of at least three competitive reactions of (I), viz. (a) oxidative fission, (b) polymerisation, with (c) (IV) either as a compolymerising unit or oxidative coupling component. J. W. D.

Melanin Formation. G. A. Swan and D. Wright. *Chem. and Ind.*, 79, (16 Jan. 1954).

Brief comment on the communications by F. K. Duxbury and Z. E. Jolles. J. W. D.

Human Visual Purple and Absolute Threshold. M. H. Pirenne. *Nature*, 173, 215-216 (30 Jan. 1954).

Criticisms are made of some conclusions by Crescitelli and Dartnall (*Nature*, 172, 195 (1953)). J. W. B.

Density of Visual Pigment in Frog Retina. G. B. Arden. *Nature*, 173, 360-361 (20 Feb. 1954).

Optical properties on exposure to light of suspensions of retinal cells from dark-adapted frogs are determined at four wave lengths, and the maximum of the difference spectrum of the visual pigment contained in the cells is found by interpolation. The volume of the cells is determined in two different ways, and results are compared and discussed. Differences between these and earlier results may be due to the pigment molecules being, as a result of certain preparative methods, more oriented, so that they absorb light more strongly when it passes down the optical axis of the cell (as in the retina *in vivo*); thus, if other absorption measurements are made with the light passing randomly, anomalies will arise. J. W. B.

PATENTS

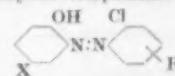
Compounds which react with Water-soluble Iron Salts to form Cyan Dyes. General Aniline.

USP 2,635,960

Heterocyclic compounds containing one of the following functional groups or a tautomer of such a group, 2-nitro-1,3-diketone or 2-isonitroso-1,3-imino-2-ketone, react with water-soluble iron salts to form cyan dyes. C. O. C.

Brown Metal(Copper)-complex Monoazo Dyes for Lacquers, etc. FBY. *BP* 704,553

o-Chloro-*o*-hydroxyazo compounds—



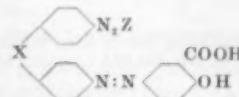
(X = Cl or Alk of 3-8 C; R = H, Cl or other substituent not causing water-solubility) are heated in alkaline alcoholic solutions of Cu compounds to yield complexes which are not water-soluble, but may be used for colouring lacquers, waxes, oils, etc. Thus the monoazo compound 3:4:8-trichloroaniline \rightarrow *p*-tert.-butylphenol is dissolved in alcoholic NaOH, a mixture of copper sulphate, glycerine and aq. NaOH is run in, and the dye formed by refluxing for ca. 7 hr. E. S.

Crimson Lakes of Monoazo Dyes for Plastics, etc. ICI. *BP* 703,845

The lakes of the monoazo dye 5-amino-2-chlorobenzoic acid \rightarrow 3:2-hydroxynaphthoic acid are colouring matters for plastics, lacquers and paints; the rosinated manganese lake in particular has good fastness to light, rubbing, migration and heat when incorporated in synthetic plastics. E. S.

Dischargeable Trisazo Direct Cotton Dyes. Gy. *BP* 704,377

The trisazo dyes obtained by coupling a diazomonoazo compound—



(X = direct link or -CO-NH-; Z = an anion; other substituents may be present but not $> 1 SO_3H$) with *N*-m- (or *p*)-aminobenzoyl-J acid, rediazotising and coupling with resorcinol are dischargeable and of good fastness to the washing-off operation. Thus tetrazotised benzidine is coupled alkaline first with 1 mol. of salicylic acid and then with 1 mol. of *N*-*p*-aminobenzoyl-J acid. The aminodisazo dye so formed is diazotised and coupled alkaline with resorcinol to yield the dischargeable bluish-red direct cotton dye. E. S.

Black Trisazo Leather Dyes. Ciba. *BP* 704,043

Trisazo dyes—

Alkaline
Acid \nwarrow H acid \longleftarrow R-NH₂

Benzidine \swarrow *m*-phenylenediamine-4-sulphonic acid

(R = Ar, and preferably contains 1 or 2 SO₃H groups) dye chrome-tanned and velour leather (suede), deep blacks, unlike dyes of the Direct Deep Black type (C.I. 581) (R = phenyl; the *m*-phenylenediamine used is unsulphonated) which give violet-blacks with a tendency to bronze. Thus the trisazo compound—

Alkaline
Acid \nwarrow H acid \longleftarrow Sulphanilic acid
Benzidine \swarrow (1) (2)

(3) \nwarrow *m*-phenylenediamine-4-sulphonic acid has excellent solubility and dyes velour leather greenish black. E. S.

1:5-Bis(*m*-fluorobenzoylamino)-anthraquinone Vat Dye. FH. *BP* 703,528

1:5-Diaminoanthraquinone is heated at 150-160°C. in *o*-dichlorobenzene, with *m*-fluorobenzoyl chloride to give a yellow vat dye of good wet and light fastness.

R. K. F.

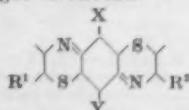
Anthrimide-Carbazole Vat Dyes. Ciba. *BP* 703,502

Polyanthrimides containing > 3 -NH- links, positioned 1:4 to each other, are treated with cyclising agents, e.g. AlCl₃-tertiary base, to produce grey vat dyes in which approx. half the original -NH- groups have been converted to carbazole rings. Thus 1:1':4':1":4":1'''4'''1'''-pentanthrimide is heated between 160°C. and 140°C. for approx. 2 hr. in a mixture of AlCl₃ and anhyd. pyridine, with simultaneous distillation of the pyridine. The

resulting dye is separated by pouring into water and purified by vatting. R. K. F.

Dithiazine Derivatives—Direct Cotton Dyes. ICI. BP 698,200

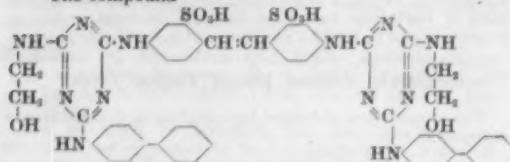
Dithiazines of general formula—



(X and Y = H, Hal or Alk; R¹ and R² = aromatic or heterocyclic), are sulphonated to give blue direct cotton dyes which are greener than the corresponding dioxazine compounds. Thus the dithiazine obtained by heating *N,N'*-di- β -naphthyl-*p*-phenylenediamine with sulphur at 200–210°C. for 16 hr. in kerosene containing a small amount of iodine, while passing a slow air current, is treated for 4 hr. at 60°C. with 10% oleum. R. K. F.

Fluorescent Brightening Agent. ICI. BP 704,974

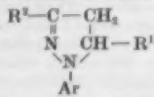
The compound—



has far higher whitening power than the stilbene derivatives mentioned in BP 624,052 (J.S.D.C., 65, 569 (1949)). C. O. C.

Pyrazolinesulphonic Acids—Fluorescent Brightening Agents. Ilford. BP 698,253

A ketone R¹-CO-CH = CH-R² is condensed with an aryl hydrazine sulphonic acid in acid medium to give pyrazoline sulphonic acids of formula—



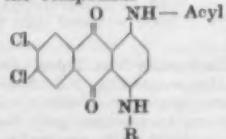
(Ar contains SO₃H; R¹ = H or hydrocarbon; and R² = hydrocarbon). The products are fluorescent whitening agents for wool. Thus, phenylhydrazine-*p*-sulphonic acid is refluxed with benzalacetophenone in dil. HCl for 5 hr. and the resulting pyrazoline separated by neutralising and adding salt. R. K. F.

Hydrolysed Cyano-anthraquinone Derivatives—Disperse Dyes. Celanese Corp. of America. BP 704,917

The 2-(and/or 3)-cyanoanthraquinones containing 1-amino and 4-substituted-amino groups of BP 639,587 and 679,589 (J.S.D.C., 66, 559 (1950) and 68, 526 (1952) respectively) are hydrolysed in the leuco form and then re-oxidised to give blue disperse dyes, the affinities of which are better than the original cyano compounds. Thus, 1-amino-2,3-dicyano-4-(β -methoxyamino)anthraquinone is heated at 70°C. for 1 hr. with 0.5% aqueous Na₂CO₃ (or potassium pyrophosphate) containing 0.5% Na₂S₂O₄, and a dispersing agent, e.g. 0.05% sodium lauryl sulphate. Both reduction and hydrolysis are thereby effected and air is bubbled through the resulting liquid to precipitate the resulting dye. R. K. F.

Dichlorophthaloylcarbazoles—Vat Dyes. Ciba. BP 704,013

Cyclisation of the compounds—



(R = an α -linked anthraquinone residue containing an α -acylamino group, and which may also be dihalogeno-substituted at 6:7) gives brown vat dyes. Thus 6:7-dichloro-4:5'-dibenzoylamino-1:1'-dianthrimide, made by

condensing 1-benzoylamino-4:6:7-trichloroanthraquinone with 5-benzoylamino-1-aminoanthraquinone, is treated with 98% H₂SO₄ for 7–8 hr. at 30°C., poured on to ice and aq. NaNO₂ slowly introduced at 0–5°C. before filtering. R. K. F.

Cobalt Phthalocyanine Vat Dyes. FB. BP 704,310

Cobalt phthalocyanines of increased hydrophilic character and suitable for applying as vat dyes, are prepared by replacing part of the *o*-arylene carboxylic acid or anhydride used in their synthesis, by an *o*-arylene carboxylic acid or anhydride carrying a $-SO_3H$, $-SO_2H$, $-SO_3NH$, $-SO_2NH$ -Alk, $-SO_3N(Alk)_2$, $-COOH$, $-CONH_2$ or $-NO_2$ group, so that the final product contains < 4 of one of such groups per phthalocyanine residue. Thus, phthalocyanine, sodium-4-sulphophthalate, cobalt sulphate, NH₄Cl, urea and ammonium molybdate are heated at 180°C. for 6 hr. in nitrobenzene and the resulting dye separated by cooling and adding methanol. Alternatively cobalt phthalocyanine is treated with oleum to give a product which is 15–40% monosulphonated.

BP 704,231

The above derivatives are applied from a NaOH-hydrosulphite vat at 50–60°C. to cotton, or from an ammonia-hydrosulphite vat at 51–52°C. to wool, to produce blue, green, and grey dyeings. R. K. F.

Non-crystallising Copper Phthalocyanine. General Aniline. BP 703,505

Copper phthalocyanine showing little tendency to crystallise, e.g. when standing for 72 hr. in toluene solution, is obtained by having present during its synthesis approx. 20% CuCl₂, 5%–7.5% AlCl₃ and 1%–2% Cl₂, all calculated on the wt. of phthalic anhydride used.

BP 703,507

Copper phthalocyanine is rendered non-crystallising by treating it in dry trichlorobenzene at 180°C., with $> 50\%$ of its wt. of AlCl₃ and with or without 1%–2% of its wt. of Cl₂, and subsequently removing the AlCl₃ by boiling with HCl. R. K. F.

Dyes. Ilford. BP 704,770

Dyes of formula R¹N-(CH₂CH₂)ₙC:(CH₂CH₂)ₘ-CR²-CY: C(CN)₂ (D = atoms to complete a 5- or 6-membered ring; R¹ = Alk or aralkyl; Y = Alk, aralkyl, Ar or heterocyclic; n and m = same or different, 0 or 1) are produced by condensing a compound of formula

Y(R¹CH₂)C:CN)₂ (R¹ [=] H or hydrocarbon; or R¹CH₂ and Y together form a carbocyclic ring) with one of

formula XR²N-(CH₂CH₂)ₙC:(CH₂CH₂)ₘ-CR²-CY: C(CN)₂ (Z = esterified carboxylic acid group) are similarly produced by replacing the first of the above intermediates by Y(R¹CH₂)C:CN)₂. C. O. C.

BP 704,840

Dyes of formula R¹N-(CH₂CH₂)ₙC:(CH₂CH₂)ₘ-CR²-CY: C(CN)₂ (Z = esterified carboxylic acid group) are similarly produced by replacing the first of the above intermediates by Y(R¹CH₂)C:CN)₂. C. O. C.

Carbon Black Pellets for News Ink. Godfrey L. Cabot. USP 2,635,057

Pelletising carbon black with a uniformly distributed surface-active constituent comprising an unsaturated fatty acid combined with an amine yields a product which disperses excellently in ordinary milling equipment and has a high degree of colour development. C. O. C.

Finely Divided Metal Oxides. Säurefabrik Schweizer-hall. BP 703,504

Volatile anhydrous chlorides, e.g. of Al, Ti or Zr, are continuously fed in fine suspension in air into a chamber where they are treated with an independently burning auxiliary flame sustained by a separate flow of combustible gas, at least some of the oxygen necessary for this flame being supplied separately from the incoming mixture. C. O. C.

Red Unhydrated Iron Oxide. Mineral Pigments Corp. USP 2,633,407

Red iron oxide is prepared without calcination by oxidising metallic iron in an aqueous mixture prepared by

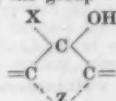
(1) adding alkali to an aqueous ferrous compound, (2) treating the resulting suspension of ferrous hydrate rapidly with a solution of a ferric compound and (3) oxidising until the mixture has pH < 3. C. O. C.

Iron Oxide Pigments. Minnesota Mining & Manufacturing Co. USP 2,634,193

High quality pigment of high tinting strength and high chrome mass tone is obtained by calcinating iron sulphate in presence of a stoichiometric amount of carbon based on the sulphate reaction according to the reaction $2\text{SO}_4 + \text{C} \rightarrow 2\text{SO}_3 + \text{CO}_2$. This reaction is exothermic and supplies an additional and internal source of heat to help to decompose the iron sulphate. C. O. C.

Pyrazolone Colour Couplers. Eastman Kodak Co. USP 2,632,702

Colour couplers are obtained by condensing a pyrazolone having a reactive methylene group in the 4-position with a compound containing the group—



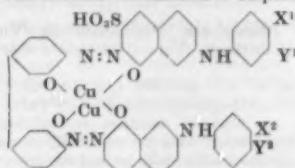
(X = H or OH, Z = atoms to complete an unsubstituted cyclic ring) so that condensation takes place between the methylene group of the first compound and a OH group of the second compound. C. O. C.

Azoic Dyes—Stabilised Diazo Compounds. General Aniline. USP 2,628,959

Precipitation of a stabilized diazo compound in the form of its chloride or sulphate, or as a double salt with ZnCl_2 etc. is effected in the presence of an *NN*-dialkyl ethylene diamine $\text{R}^1\text{CONH}_2\text{C}_2\text{H}_4\text{N}(\text{R}^2)_2$ (R^1 = alkyl of 12-18 C; $\text{R}^2 = \text{CH}_3$ or C_2H_5) resulting in a product of improved crystalline form and filtering properties. E. S.

Grey Direct-cotton Metal(Copper)-complex Disazo Dyes. DuP. USP 2,630,431

The copper complexes of disazo dyes derived from tetrazotised dianisidine and suitable *N*-sulphonyl- γ -acids—



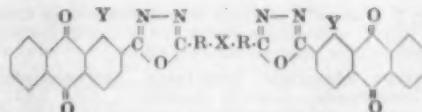
(at least one of X^1 , Y^1 , X^2 and $\text{Y}^2 = \text{SO}_3\text{H}$; the others = H or CH_3 ; not more than one CH_3 and one SO_3H is attached to the same aryl radical) are direct cotton greys of good fastness to light and crease-resist finishing processes. Thus the disazo dye dianisidine $\xrightarrow{\text{N-}p\text{-sulphonyl-}\gamma\text{-acid}}$, is heated with aq. ammoniacal copper sulphate to give a blue-grey direct cotton dye. E. S.

Black Tetrakisazo Direct Cotton Dyes. CFM. BP 703,446

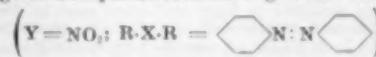
The tetrakisazo dyes obtained by tetrazotising the diaminodisazo compound 4:4'-diaminodiphenylamine-2-sulphonic acid $\xrightarrow{\text{2-amino-8-naphthol-6-sulphonic acid}}$, and coupling with 1 mol. of *m*-phenylene (or toluylene) diamine (which may be *N*-hydroxyethylated) and 1 mol. of a naphthol, naphthylamine or aminonaphthol, free of SO_3H and COOH groups, dye cellulose fibres bluish and violetish blacks instead of the greenish blacks produced by coupling the same tetrazo compound with 2 mol. of *m*-phenylenediamine or its *N*-hydroxyethyl derivatives as described in BP 470,407 (J.S.D.C., 53, 488 (1937)). Thus by coupling with 1 mol. of *m*-phenylenediamine and 1 mol. of 1-amino-8-naphthol in alkaline solution a dischargeable bluish-black is produced. E. S.

Red Anthraquinone Azo Vat Dyes. DuP. USP 2,628,230-1

1-Amino (or nitro) anthraquinone-2-carbonyl hydrazines are reacted with suitable organic dicarboxylic acid chlorides and the products ring-closed to oxadiazoles—



(Y = NH_2 or NO_2 ; R = a benzene or diphenyl radical; X = $-\text{N}(\text{H})_2$, $-\text{CH}=\text{CH}_2$, $-\text{CH}_2-$, O, S, $-\text{SO}_3-$ or $-\text{CO}-$) which are red vat dyes. Thus 1-nitroanthraquinone-2-carbonyl hydrazine is heated in nitrobenzene with azobenzene-4:4'-dicarbonyl chloride, and the product ring-closed by heating with SOCl_2 in nitrobenzene to give the oxadiazole—



During the vatting process the NO_2 groups are reduced to NH_2 . Or the NO_2 groups may be replaced by NH_2 by treatment with NH_2 in nitrobenzene or in an autoclave, to give a scarlet vat dye. E. S.

1-Aminoanthraquinone. American Cyanamid Co. BP 703,678

Sodium or potassium anthraquinone-1-sulphonate is heated with a small molecular excess of 3-8% aq. NH_3 at 150-180°C. (max. pressure = 350 lb/sq. in.) for 8-15 hr. in presence of an oxidising agent, e.g. sodium nitrobenzenesulphonate, and with or without boric acid (25-33% of the wt. of the anthraquinone sulphonate) to give unspecified yields of 1-aminoanthraquinone of m.p. 253-254°C. R. K. F.

Indanthrone from 1-Aminoanthraquinone. FBy. BP 703,577

Indanthrone is made from 1-aminoanthraquinone by heating it with not more than four times its wt. of alkali phenolate containing an oxidising agent, e.g. NaClO_4 and the alkali metal salt of a fatty acid having < 5 C atoms. Thus, all parts being by wt., 1-aminoanthraquinone (100) and NaClO_4 (5-20) are added to a molten mixture, of KOH (100), phenol (150) and anhyd. sodium acetate (40) at 160-180°C. Fe_2O_3 (1) or MnO_2 (1) is next introduced and the temp. increased to 210°C. After diluting with water, filtering and drying, the crude product is purified by dissolving in conc. H_2SO_4 , diluted to 83% H_2SO_4 and filtered to give 68.2 parts pure dye. Unchanged 1-aminoanthraquinone (9.6) is recovered from the filtrate. R. K. F.

Halogenated Dibenzanthrones, isoDibenzanthrones and Dibenzanthrynes; and Condensation Products thereof with Aminoanthraquinones

General Aniline. BP 703,296

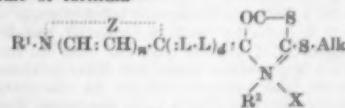
Dibenzanthrone, isoDibenzanthrone and dibenzanthrynes are treated with a halogen in H_2SO_4 or chlorosulphonic acid containing 25-60% of the wt. of substance being halogenated or anhyd. aluminium halide. The products differ from those obtained by the usual procedures using sulphur or iodine, instead of aluminium halide, as carrier. Thus bromine is added to a soln. of dibenzanthrone in chlorosulphonic acid containing AlCl_3 in suspension, at 25°C. and stirred for 24 hr.

BP 703,294

The above products are condensed with aminoanthraquinones and the resulting anthraquinonyl amino compounds cyclised to give navy blue to black vat dyes. Thus the bromo derivative obtained in the example quoted above is refluxed with 1-aminoanthraquinone, Na_2CO_3 , CuCO_3 , and CuO in nitrobenzene. After separating, the resulting product is heated at 150-155°C. for 1 hr. with alcoholic KOH, diluted with water and oxidised with air. R. K. F.

meroCyanine Dyes. Kodak. BP 703,526

meroCyanine dyes are obtained by condensing a quaternary salt of formula—



with a primary amine of formula R^3NH_2 (R^1 and R^2 = same or different, subst. or unsubst. Alk; R^3 = subst. or unsubst. hydrocarbon, or heterocyclic

VI—FIBRES; YARNS; FABRICS

Static Electrification of Textile Yarns—L. V. E. Gonsalves. *Text. Research J.*, 23, 711–718 (Oct. 1953).

The problem of static electricity in textiles is discussed, and a theory put forward to explain its production, with particular reference to the case of an insulator rubbing against a metal.

A. B.

Discharge of Electrified Textiles. J. A. Medley. *J. Textile Inst.*, 45, T123–T141 (Feb. 1954).

Discharge processes, which may be utilised for the dissipation of static, are analysed quantitatively. They are of three main types—(1) conduction within the material, which is responsible for the humidity effect; (2) conduction by surface impurities, a different mechanism which accounts for the action of antistatic agents; and (3) gaseous discharge, which determines maximum charge densities in accordance with geometrical considerations. A special drafting roller is described, made of porous cast-iron material, which is impregnated with a suitable conducting lubricant. A negligible film—less than 0.02% on wt. of wool—is produced on slivers passing through, but is quite efficacious in removing static. The function of the agent is to provide conduction from the fibre to the roller at the point of nip, and not to conduct electricity back to the point of contact, because such a treated sliver is electrified strongly if subsequently passed through ordinary rollers.

J. W. B.

Abrasion Resistance of Fabrics when Test Units of Abrasion are combined with Test Units of Laundering, Dry-cleaning, and Light. M. O. Zook and F. B. Mack. *Amer. Dyestuff Rep.*, 43, 61–66 (1 Feb. 1954).

A preliminary study is made of the relative changes in strength and colour which result from abrasion alone and abrasion combined respectively with the agencies of light, laundering, and dry cleaning, in different orders of application. It appears that other agents applied alternately affect the abrasion resistance in various ways, depending on the characteristics of the fabric, and that a different final resistance to abrasion is given from that obtained by abrading new fabrics alone. Only by combined tests is it possible, therefore, to predict in the laboratory the resistance under actual use conditions.

J. W. B.

Spoilage of Industrial Materials by Micro-organisms. T. Richards. *Nature*, 173, 102–104 (16 Jan. 1954).

A symposium on microbial damage in industrial materials is surveyed. Damage to paints, textiles, and paper, and methods of prevention, are discussed.

J. W. B.

Fluorescence Changes in Deltapine-15 Seed Cotton as a result of heat. W. C. Sheehan, T. L. W. Bailey, and J. Compton. *Text. Research J.*, 23, 736–743 (Oct. 1953).

It is shown that the fluorescent colour of untreated cotton in ultraviolet radiation changes from bluish white to ivory white on heating. This is similar to the effect reported by investigators for the ageing of cotton.

A. B.

Ultrasonic Treatment of Lint Cotton. A. E. Martin. *Text. Research J.*, 23, 727–730 (Oct. 1953).

The effects of ultrasonic radiation on lint cotton have been investigated, and at the frequencies and energy densities employed no mechanical damage was induced. Evidence of crystallinity change was present to a small degree.

A. B.

Structures of Native and Mercerised Celluloses. M. K. Sen and S. C. Roy. *Nature*, 173, 298–300 (13 Feb. 1954).

Previously reported new equatorial reflexions in the diffraction pattern of native celluloses of various origins (M. K. Sen and H. J. Woods, *Biochim. Biophys. Acta*, 3, 510 (1949)) are further studied. It is found that one reflexion, γ , is common to almost all ordinarily available cellulose fibres, native or mercerised. At 60–70% R.H. and 70–75°F. the sharpness, intensity and dispersion of the diffraction maximum are at least equal to those of certain other equatorial reflexions. It is concluded that the reflexion originates from the cellulose crystallites, the difference in intensity and the variation in spacing for different samples arising from the formation of mixed crystals with other polysaccharides and/or adsorption of

water, which may be water of hydration or structural water. Parameters are calculated of unit cell structures into which all reflexions from native and mercerised ramie can be fitted; with a slight shift other cellulose fibres can be accommodated. It is considered that previous unit cells do not represent the structure of the material under ordinary conditions of temperature and humidity. The theory of the impenetrability of cells of native cellulose by water does not seem to be tenable, and it appears that the current concept of the crystalline and non-crystalline regions may need revision. Reasons are appended for the failure of previous workers to observe the new reflexion.

J. W. B.

Preparation of Soluble Yarns by the Carboxymethylation of Cotton. G. C. Daul, R. M. Reinhardt, and J. D. Reid. *Text. Research J.*, 23, 719–726 (Oct. 1953).

Water- and alkali-soluble cotton fibres have been prepared by pressure-kiering in 2% NaOH to remove the non-cellulosic materials, and then treating with chloroacetic acid and NaOH by either a one-step or a two-step process. The treated cotton is neutralised in alcohol with acetic acid to produce water-soluble products or with strong acids to produce water-insoluble but alkali-soluble products.

A. B.

IV—Researches by Chromatography—Impurities of Cotton and Wood Cellulose. A. Hatano and H. Sobue. *Bull. Chem. Soc. Japan*, 26, 403–406 (Sept. 1953).

The sulphuric-acid hydrolysates of raw and purified Egyptian cotton linters, Japanese red pine (*Pinus densiflora*) and Japanese beech (*Fagus crenata*) on paper chromatography with *n*-butanol-acetic acid-water (4:1:1) yield the following compounds in addition to glucose: Purified linters—traces of arabinose and glucuronic acid. Raw linters—1.5% arabinose, small amounts of galactose, glucuronic acid, and galacturonic acid, probably derived from pectin. Raw linters, pine, and beech—cytine, aspartic acid, asparagine, arginine, glutamic acid, alanine, and tryptophan, corresponding to a total protein content of 0.2–0.3%. Pyrrolidonecarboxylic acid is also produced by re-arrangement of glutamic acid.

A. J.

Association of Xylan with α -Cellulose in Jute. A. K. Mazumdar and P. B. Sarkar. *Nature*, 172, 1047–1048 (5 Dec. 1953).

Purified α -cellulose from jute, hydrolysed with 85% formic acid at 135°C. for 12 min., leads to a chromatogram on which no xylene or arabinose can be detected, which disagrees with earlier results by Das *et al.* (*Nature*, 171, 613 (1953)). Experimental technique and purity of the α -cellulose are suggested as being at fault, and it is stated that under those conditions xylene is not more resistant to formic than to sulphuric acid.

J. W. B.

Addition Reaction of Phenyl isoCyanate with Bleached Ramie. P. Schneebeli. *Assoc. tech. ind. papetiere Bull.*, 1953, 223–9; *Chem. Abs.*, 48, 1673 (10 Feb. 1954).

The progress of the addition reaction between phenyl iso-cyanate (I) in dry pyridine at 100°C. and commercially bleached ramie (II), laboratory bleached ramie (III) and purified cotton linters (IV) was followed chemically and microscopically. Submicroscopic studies of the progressive addition of I to OH groups in cellulose were made by noting the changes in X-ray diffraction patterns and those in water-vapour sorption. The degrees of polymerisation of II, III, and IV were 1275, 1315, and 880 respectively. N determinations showed that all OH groups in II reacted with I within 4 hr. and in IV within ca. 12 hr. Esterification of III was incomplete even after 16 hr. unless III had previously received 48 hr. treatment with I in pyridine at room temperature (this caused no actual addition of I). Initial reaction of I with II or III took place at the nodes (i.e. "slippage planes"). When all the OH groups had reacted, the products were gels that still retained some structure of the original fibres. No change in X-ray diagrams appeared until about 28% of the OH groups had been covered. In the initial phase of I addition (in II, III, and especially IV) the water-vapour sorption decreased. This is because the readily accessible OH groups, which give the fibres their hydrophilic properties, are the first esterified.

C. O. C.

Nitration and Fractionation of Rayons. W. E. Roseveare and L. Poore. *Ind. Eng. Chem.*, **45**, 2518-2521 (Nov. 1953).

Serious difficulties were encountered in applying to rayons the published procedures for the nitration and fractionation of cellulose. Various nitrating methods are compared, and a suitable procedure for preparing a stable undegraded cellulose nitrate from rayon, is described. Two-step fractionation of the cellulose nitrate was found to have little advantage over one-step fractionation except in separating more material of very low and very high degree of polymerisation by the refractionation of the lowest and highest one-step fractions. W. K. R.

Cross-linking in Cellulose Fibres. I—Physical and Mechanical Properties of Cellulose Fibres treated with Diisocyanate. M. Kamiya and Y. Zyo. *J. Soc. Textile and Cellulose Ind., Japan*, **9**, 469-474 (1953); *Chem. Abs.*, **48**, 1671 (10 Feb. 1954).

Viscose rayon purified successively with ethyl alcohol and ether and heated with 0.5% soln. of tetramethylene diisocyanate in benzene at 80-120°C. when the N was distributed uniformly, swelled less in water when the N content was higher, reaching a limit with 0.4% N (one cross link/400 glucose units). The solubility in alkali decreased similarly, with the N limit somewhat higher. The tensile strength and elongation were also affected somewhat by the N content. Wet strength increased markedly with 0.4% N and elongation reached maximum at this point. C. O. C.

Plasticity of Wool. II—Variation with Type of Follicle. R. W. Burley and J. B. Speakman. *Text. Research J.*, **23**, 702-710 (Oct. 1953).

It is shown that the variations in plasticity among the fibres of single staples of New Zealand Lincoln, New Zealand Romney, and Australian merino wools are associated with follicles of different types and ages. Primary follicles produce fibres of lower plasticity than those of secondary follicles, and the order of plasticity of the fibres is, in general, the inverse of the order of development of the follicles. A. B.

Apparel Wools. IV—Physical Properties of Single Fibres. T. F. Evans and D. J. Montgomery. *Text. Research J.*, **23**, 689-701 (Oct. 1953).

The physical properties of single wool fibres have been determined at various stages of worsted processing. The following properties were taken from the force-extension curve—slope in the Hookean region, force at 20% extension, energy for 20% extension, force at break, and extension at break. These were then related to the cross-sectional area. The changes in the force-extension properties during processing were found to be definite, but not great. A. B.

Frictional Properties of Wool Fibres. B. Lincoln. *J. Textile Inst.*, **45**, T92-T107 (Feb. 1954).

Frictional properties of the wool fibre are discussed in terms of the geometry of an ideal scale profile and on the assumption that the deformation of the material in regions of true contact is substantially elastic. A general relationship between elastic properties, location of contact on the scale, and frictional force is developed and enables frictional forces to be calculated for a wide range of geometrical and elastic conditions. Prediction of relationship between frictional force and load, and the variation of friction with regain, give good agreement with published data. Accurate conclusions concerning the nature of deformation undergone by the scale material at the regions of true contact cannot, however, be made from existing experimental data. The effects of fibre quality are studied and it is shown that the frictional properties of wool vary with the distribution of scales rather than with fibre diameter. Mohair, in which scale dimensions are less dependent on fibre diameter, exhibits properties which vary less markedly with fibre quality. It is suggested that dry chlorination modifies scale surface rather than scale edge forces, a conclusion supported by effects associated with lubrication of such fibres. J. W. B.

Purification of Wool. J. P. E. Human and J. B. Speakman. *J. Textile Inst.*, **45**, T162-T165 (Feb. 1954).

It is shown that extremely prolonged solvent extraction is necessary to purify raw wool, and an ingenious modification of the conventional Soxhlet is described which

enables extractions lasting 50-100 hr. to be carried out without loss of solvent through leakage. Even after 1,000 hr. detectable traces of wax can still be extracted from the wool. Load-extension curves of normally and exhaustively purified wool are determined to see whether residual wax has any effect. Work to 30% extension is unaffected, but the breaking load appears slightly lower with normally extracted fibres, though results are not statistically conclusive. All the long-extracted fibres give the Allwörden reaction with bromine water. J. W. B.

Selective Absorption of Optical Antipodes by Proteins. W. Bradley and R. A. Brindley. *Nature*, **173**, 312-313 (13 Feb. 1954).

Wool is immersed in a solution of (\pm) - α -methoxyphenyl acetic acid in a mixture of equal vol. of water and alcohol, and after a time the amounts of unadsorbed and adsorbed acid, and the rotations of the two fractions, are determined. They are of the same order as those previously obtained with mandelic acid in similar circumstances, and factors enabling the resolution of acids on proteins to be demonstrated are discussed. The protein must carry an adequate number of basic groups (mandelic acid is resolved on wool and casein but not on silk); the molecular rotation of the acid should be high, as the degree of separation so far observed is small; and the acid should not contain groupings which favour the non-specific absorption of its amino by non-ionic forces; e.g. *p*-hexadecoxymandelic acid is not resolved, and many acids containing azo groups are not resolved on wool. The resolution, and the extent to which it occurs, are of considerable interest for the theory of biochemical processes. J. W. B.

Protein Structure. J. C. Kendrew. *Nature*, **173**, 57-59 (9 Jan. 1954).

In a symposium on protein structure, current theories are reviewed and discussed, including those relevant to silk and collagen. J. W. B.

Acetate Blends of Improved Performance. F. Fortess, B. S. Sprague, and R. G. Stoll. *Amer. Dyestuff Rep.*, **42**, P 851-P 864 (7 Dec. 1953).

Data are presented on acetate-viscose rayon and acetate-viscose rayon-nylon blends having optimum performance characteristics, and although the fabrics concerned are for sport shirtings and other light-weight wear, the techniques discussed for assessing blend-serviceability relationships may be applied to all types of material. Suitable finishing procedures for improving performance are suggested. The construction, dyeing procedure, and performance characteristics of acetate-wool blends are described, and the effectiveness of acetate rayon in giving wool-like performance is emphasised. J. W. B.

Influence of Various Classes of Dyes on the Strength of Nylon on Exposure to Light. C. Zuber, J.-P. Niederhauser, J. Gobet, and J. Chezard. *Tintex*, **19**, 3-13 (Jan. 1954).

The effect of a number of dyes from various groups on nylon when exposed to light was examined. Individual acid, disperse, and direct dyes may exert a favourable or adverse effect, but no general conclusions could be drawn. Vat dyes, especially yellows, oranges, and reds have a distinctly adverse action, which is most likely akin to similar behaviour observed on cotton. Chrome and pre-metallised (Inochrome) dyes have a protective action, and the results with the former confirm the work of Newsome on logwood. B. K.

Solubility Diagrams for the Ternary System consisting of ϵ -Caprolactam, Water, and (a) Dichloroethane, (b) Chloroform, (c) Methylene Dichloride. G. I. Kudryavtseva and A. D. Krutikova. *J. Appl. Chem. U.S.S.R.*, **26**, 1190-1194 (Nov. 1953).

Cyclic Oligomers of Caprolactam (Unusual Case of Stereo-isomerism in Higher-membered Rings). P. H. Hermans. *Rec. Trav. Chim.*, **72**, 798-812 (Sept.-Oct. 1953).

The low-mol wt. substances other than caprolactam, present in hot-water extracts of caprolactam polymerates with an average degree of polymerisation of ~ 150 , are investigated, and three individual compounds isolated, none of which contains free COOH or NH₂ groups; they are cyclic oligomers of the lactam, two being cyclic dimers

and the other a cyclic trimer. The possible nature of this unexpected isomerism is discussed. H. H. H.

Application of Glass Fibres in Filtration Processes.
C. A. Smucker and W. C. Marlow. *Ind. Eng. Chem.*, **46**, 176-178 (Jan. 1954).

The resistance of glass fibre to heat and chemicals makes it valuable in many filtration processes. Illustrations are given of the versatility of glass fibre materials as filtration media, achieved by the combination of various fibre sizes, structures, and bonding materials. W. K. R.

PATENTS

Chemical Retting of Vegetable Fibres. Société d'Exploitation des Procédés Lourd S.E.P.L. *BP* 703,666

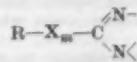
Flax, hemp, jute and similar fibres are retted by subjecting them firstly to an aqueous alkali, e.g. 0.5%-2% NaOH, containing the palmitate of an amine base which acts as an emulsifier, and then to a second solution similar to the first but also containing an oxidising agent. J. W. B.

Removing Hair and Wool from Hides and Skins.
K. Wolf. *BP* 704,569

The hair and wool can be readily removed from any sort of hide or skin by use of a water-insoluble alkaline earth metal sulphide evenly loaded into paper, if the skin is first treated with an aqueous solution of an agent, e.g. sodium sulphate or carbonate, which reacts with the alkaline earth metal sulphide to form a soluble sulphide and an insoluble alkaline earth metal compound. C. O. C.

Reduction of Static in Yarn, Thread and Filament Processing. Shell Refining and Marketing Co. *BP* 703,499

Electrostatic charges on textile yarns are reduced by applying, before or during processing, an organic compound free from sulphonate acid groups and containing the grouping—



(R = organic radical; X = O, S or imino; m = 0 or 1). It may be applied dissolved in a mineral lubricating oil which also contains an oil-soluble compound containing an alcoholic hydroxyl group. In one example solvent refined mineral lubricating oil (89.5 parts by wt.) is mixed with 1-β-hydroxyethyl-2 hepta-decyleneimidazoline (2.5), glyceryldioleate (5.0) and nona-ethyleneglycol mono-ooleate (3.0). This blend spontaneously emulsifies when diluted to 10% with tap water and is readily scoured from the yarn. J. W. B.

Acrylonitrile-Polyamine Copolymers having Affinity for Acid Dyes. Eastman Kodak Co. *USP* 2,632,748

Compositions containing polyacrylonitrile (70-90 parts by wt.) and a polyamine containing amide groups (30-10) yield clear stable homogeneous solutions in common volatile organic solvents. Such solutions can be either dry or wet spun to yield fibres which are readily dyed with acid dyes. C. O. C.

Acrylonitrile Copolymers having Good Affinity for Acid Dyes. American Cyanamid Co. *BP* 705,887

Copolymers of acrylonitrile with compounds of formula—



R¹ and R² = same or different, H or CH₃; R³ and R⁴ = same or different, H or Alk; n = 1 or 2) are more readily dyed, especially with acid dyes, than polyacrylonitrile or many of the known copolymers of acrylonitrile. C. O. C.

Alkali or Alkaline Earth Metal Salts for lowering the Viscosity of Polyacrylonitrile Solutions.
Chemstrand Corp. *USP* 2,632,750

Addition of sodium thiocyanate, lithium bromide, calcium chloride or magnesium bromide to organic solvent solutions of acrylonitrile polymers lowers the viscosity and increases the fluidity of the solutions. C. O. C.

Plastic and Textile Materials. O. M. Arnold.

USP 2,636,014

When a synthetic organic fibre-forming linear superpolyamide, e.g. nylon, is intimately associated with a cellulosic plastic, e.g. cellulose acetate, and then heated and pressed the product is very suitable for making shaped-plastic articles including fibres. Such a mixture is less liable to deterioration by oxidation even at softening temperature and is in general more heat-resistant than is nylon by itself. C. O. C.

Coated Threads. North British Rayon Ltd. *BP* 702,828

Metallic or non-metallic threads are coated with resins by drawing them through a bath of the molten resin at such a speed that a certain quantity of resin adheres and, the thread being drawn away vertically, tends to run backwards down it so that it collects in globules, thus forming an irregular coating. Applying vibration can be used to assist, either in the immediate drawing or in subsequent passage over a doctor blade when the beads have hardened. Such fibres have uses as novelty threads or, in the case of coated metal, in determining the spacing between wound wires, the spacing depending on the size of the globules.

BP 702,829

Detailed operational conditions are given for the production of irregularly-coated threads, with examples.

J. W. B.

Fabrics consisting of Adhesively-bound Threads.

British Cotton Industry Research Association. *BP* 703,614

Longitudinal threads are continuously advanced as layers against flat faces of a stationary former. Transverse threads are laid across the outer face of each layer of longitudinal threads by winding at least one continuous thread around the form. Direct pressure is applied to the outer face of the transverse threads so as to cause the longitudinal threads to be pressed slidably against them, heat being applied at the same time. The threads may contain a thermoplastic adhesive or such an adhesive may be applied to the outer face of the layer of longitudinal threads immediately before laying the transverse threads. C. O. C.

Production of Pile Fabrics by Electrostatic Deposition. Dunlop Rubber Co. *BP* 704,456

In a machine where the adhesive-coated backing is passed between two electrodes, the backing touching the upper electrode and having its coated side downwards, and a conveyor belt carrying pile-forming fibres passed in contact with the lower electrode, more pile fibres per unit area are fixed to the backing if the latter is vibrated, preferably at 5,000-15,000 cycles per min., as it passes between the electrodes. C. O. C.

α- and *β*-Forms of Poly-L-alanine (XII p. 213). Catalytic Action of Organic Peroxides on the Polymerisation of Ethenoid Compounds. II—Activity of Substituted Benzoyl Peroxides on the Polymerisation of Vinyl Acetate (XIII p. 214).

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

PATENTS

Removing Mineral Oil from Vegetable Fibres with Simultaneous Flameproofing of the Fibres.
Lockport Cotton Batting Co. *BP* 704,460

The fibres are soaked in aqueous medium, preferably containing borax or boric acid, hydroextracted until they contain 45-50% of their dry weight of the solution and are then dried to 30% water content by air at 120-160°C., the water content then being reduced to 15% with air at 90-120°C., drying being finally completed at < 100°C. This volatilises the mineral oil with the water and leaves a flameproofed material. C. O. C.

Bleaching Vegetable Fibres. S. A. Geernert & Matthys Frères. *BP* 704,111

If the fibres are first treated for a few minutes at 50°C. with a 0.5% aqueous alkyl ester of an aryl monosulphonic

acid, e.g. nonyl ester of naphthalene monosulphonic acid, subsequent bleaching with chlorine is much facilitated.
C. O. C.

Chemistry of Chelation in Textile Processing (III p. 194). Desizing of Glass Fibre Fabrics and their subsequent Coating (X p. 211).

VIII—DYEING

New Problems in Garment Dyeing. B. Kramisch and F. Grainger. *Dyer*, 110, 751-757 (13 Nov. 1953).

Dyeing procedures are described for mixtures of wool, cotton, linen, acetate rayon, and silk with many of the newer fibres. Redyeing of faded wool garments is discussed, and the Cibalane are said to be suitable for covering irregularities due to exposure to light. Albatex PO is recommended as a levelling assistant for union dyes when applied to faded materials. A. H.

Dyeing of Cellulosic Fibres. H. R. McCleary. *Text. Research J.*, 23, 673-682 (Oct. 1953).

The theoretical aspects of the dyeing of cellulose with substantive dyes are reviewed. The amorphous and crystalline regions of cellulose are discussed in relation to the dye uptake of the fibre. The mechanism of diffusion and the effect of sorption on diffusion are detailed, together with the determination of affinities of dyes for cellulose. It is shown that the skin structure of rayons slows down the penetration of dye into the fibre. A. B.

Absorption of Chrysophenine G by Cotton and by Acidic Oxycelluloses. M. M. Bilsbury, J. T. Martin, and H. A. Standing. *J. Textile Inst.*, 45, T1-T20 (Jan. 1954).

The equilibrium absorption of Chrysophenine G by cotton cellulose and by carboxyl-containing oxycelluloses prepared from it is studied. Introduction of carboxyl groups into the cotton cellulose by oxidation results in a lowering of dye absorption from solutions at pH 7 but not at pH 3, the effect being greater at low than at high salt concentration. At constant pH and salt concentration the absorption-dye concentration relation fits the Freundlich equation, and the exponent n does not increase significantly with increasing carboxyl content of the cellulose. For cotton the relation between salt:dye ratio and substantivity ratio is almost independent of the dyebath composition over a limited range of salt concentration. As predicted by theory, for acidic oxycelluloses the substantivity ratio at a constant salt:dye ratio decreases considerably with decreasing salt concentration. Measurements with viscose sheet, which may be regarded as a regenerated acidic oxycellulose, show that the relation between salt:dye ratio and substantivity ratio depends partly on the degree of swelling of the sheet. J. W. B.

Direct Dyeing of Cellulose—A Method for the Analysis of Equilibrium Absorption Data in Terms of Current Quantitative Theories. H. A. Standing. *J. Textile Inst.*, 45, T21-T29 (Jan. 1954).

Equations derived from current theoretical treatments of the equilibrium between cellulosic materials and direct dyes are reduced to the same form, which expresses a unique relation between the substantivity ratio q and the salt:dye ratio p ; it is called the p, q equation and contains two empirical constants A and B —

$$Aq^{\frac{1}{p}} + pq - B(p + 1) = 0$$

which may under commonly occurring conditions be approximated to—

$$q = -\frac{Aq^{\frac{1}{p}}}{p} + B$$

which requires a straight line of slope $-A$ and intercept B in a plot of q against $q^{\frac{1}{p}}/p$. This is convenient for testing the fit of experimental absorption data to the quantitative theories. Values of A and B are readily determined from suitable data that fit the p, q equation. The constant that has been defined as the volume of water in litres associated with 100 g. of cellulose and acting as a solvent for the unabsorbed ions in the ionic cloud surrounding the dye anions is symbolised by V , and is here given units of

litres kg. A method is given for the quantitative determination of V according to whether the cellulose is native, mercerised, or regenerated; it is shown that—

$$V = \frac{\sqrt{B}}{A}$$

An attempt is made to fit data for the absorption of Chrysophenine G by cotton and mercerised cotton to the p, q equation, but except as an approximation and over a limited range of salt concentration, no fit is obtained with the data for cotton. Calculation of V , and also of thermodynamic affinity $-A\mu^{\circ}$ from the equation—

$$-A\mu^{\circ} = RT \ln AV/B$$

is therefore not possible on the basis of these theories. They also fail to correlate available data for the absorption of the dye at 90°C. by mercerised cotton with those available for its absorption by non-mercerised cotton at the same temperature. J. W. B.

Azoic Coupling Components: Relationship between Chemical Structure and Substantivity for Cellulose. J. Organic Chem., 18, 1292-1316 (Oct. 1953).

The following relationships have been shown to exist between the substantivity of acylamides of aromatic α -hydroxy carboxylic acids for cellulose and their spatial arrangement.

- (1) Products with linear configuration have better substantivity than angular types.
- (2) Flat or coplanar products have better affinity for cotton than analogous or isomeric products which are non-coplanar.
- (3) Substitutions or modifications which disrupt or disturb the coplanar configuration of the parent nucleus result in reduced substantivity.
- (4) Minimum fixation on cotton occurs with compounds which are predominantly linear and coplanar in configuration and which contain several substantive groups 10A or multiples thereof apart, corresponding to the periodicity of the cellulose units which make up the cellulose chain.

These relationships are similar to those observed for direct cotton dyes and agree with the conception that coordinated hydrogen provides the link between cellulose and the direct dye or the coupling component.

C. O. C.

Effect of Urea-Formaldehyde Resins on the Light Fastness of Direct Cotton Dyes. A.A.T.C.C. Rhode Island Section. *Amer. Dyestuff Rep.*, 43, P6-P3 (4 Jan. 1954).

As the effect of urea-formaldehyde aftertreatment on direct dyes varies according to the dye and cannot be predicted, the following factors are studied—molecular structure of the dye; possibility of reaction between resin and dye; possibility of resin breakdown under the influence of light, with the formation of a product that affects the dye; urea/formaldehyde molar ratio; curing catalyst; and pH of the resin-finished fabric. Reactive groups in the dye molecule appear to be OH and NH₂, but although their location has some influence on the effect of resin, no definite conclusion is possible. Treatment with formaldehyde + catalyst reduces the light fastness of direct dyes with $-\text{NH}_2$ groups attached to the naphthalene nucleus adjacent to the azo groups, but different results may be obtained with urea-formaldehyde + catalyst, and different again with hydroxymethylurea + catalyst. Increasing the molar ratio of formaldehyde to urea in the resin increases the degrading effect on light fastness, and a less harmful effect is caused by non-ammoniacal catalysts. It is possible to make predictions with some dyes, but no general rule can be formulated. J. W. B.

Direct Cotton Dyes developed with Copper Salts. P. Disorens. *Teintex*, 18, 683-702 (Nov.); 769-777 (Dec. 1953).

A useful summary of the chemistry of copperable direct dyes together with patent references and structural formulae. B. K.

Structure of Viscose Rayons and the Causes of "Barry" Dyeing. J. Meybeck, N. Iwanow, and R. Schneider. *Teintex*, 18, 747-769 (Dec. 1953).

Experiments are described which examine the influence of denier, drawing, steaming, and oxidising agents on the

dyeing properties of "barry" viscose rayon and also some factors, such as salt concn., which affect the penetration of dyes into the fibre.

B. K.

Dyeing of Acetate Rayon. VIII—Effect on Dyeing of Partial Saponification (1) and (2). R. Tanaka and K. Seko. *J. Soc. Textile and Cellulose Ind., Japan*, 9, 127-132 (1953); *Chem. Abstr.*, 48, 1688 (10 Feb. 1954).

(1) Acetate rayon partially saponified for 1-30 min. at 20-50°C. with 0.5-1.6% NaOH and dyed with Nippon Sky Blue 390% showed greater affinity for the dye with lesser acetyl values down to 47.8% below which the affinity no longer increased markedly. Addition of a neutral salt, e.g. Na tartrate, to the dyebath checked saponification and also prevented damage to the fibres.

(2) Acetate rayon saponified to 47.8% acetyl value has an outer layer of regenerated cellulose of thickness 5% of the radius of the unsaponified fibre. When a section was dyed with Nippon Sky Blue 390% and Celliton Red R on a slide, the saponified peripheral layer, about 7000 Å thick, dyed blue and the unsaponified dyed red. Owing to mechanical stirring saponification was not uniform, but the thickness of the saponified layer made the surface look smooth without spotty dyeing.

C. O. C.

Fastness Properties of Acetate Rayon Dyeings aftertreated with Curable Resins. A.A.T.C.C. Fairleigh Dickinson College Student Chapter. *Amer. Dyestuff Rep.*, 43, P 50-P 52 (18 Jan. 1954).

As little is known about the effect of curable resin finishes on disperse-dyed acetate rayon in acetate-viscose rayon blends, a study is made of the effect of urea-formaldehyde, melamine-formaldehyde, and of a water-repellent finish with added urea-formaldehyde, on a delustered acetate crepon, which is then exposed to light and gas fumes. It is concluded that urea-formaldehyde has a slightly adverse effect on light fastness, and melamine-formaldehyde also except with yellows, which are improved. In general, urea-formaldehyde has a pronounced adverse effect, and melamine-formaldehyde no effect, on gas fading. The water-repellent plus urea-formaldehyde finish is slightly more beneficial to gas fading and also improves light fastness considerably.

J. W. B.

Dyeing Solid Colours on Blends of Dacron or Orlon with Wool. A.A.T.C.C. Clemson College Student Chapter. *Amer. Dyestuff Rep.*, 43, P 48-P 49 (18 Jan. 1954).

Blends of 55% synthetic fibre (Dacron or Orlon) and 45% wool are vat-dyed by padding in mixture of dye and acrylonitrile-butadiene latex and drying, followed by reduction of the dye, rinsing, oxidation, and soaping. The latex fixed in drying permits better fixation of the vat dye on the blend during subsequent reduction and oxidation, leading to improved fastness to light, washing, dry cleaning, and rubbing. In some instances improvement is effected by adding cationic agents to the latex-dye pad liquor. Control of colour, to prevent the wool from taking most of it, may be assisted by controlled addition of a lignosulphonate. For cross-dyeing, the method may be varied by soaping the pigment from the wool before dye-fixation, and subsequently dyeing the wool with any suitable fast dye.

J. W. B.

Dyeing and Processing of Synthetic Fibres. R. A. Travis. *Amer. Dyestuff Rep.*, 42, 845-847 (7 Dec. 1953).

A brief survey of the general characteristics and dyeing properties of Terylene, Orlon, dynel, and Acrilan.

J. W. B.

Dyeing of Hydrophilic Synthetic Fibres. Dyer, 111, 47-48 (8 Jan. 1954).

The structures of natural fibres, hydrophilic synthetic fibres, and hydrophobic synthetic fibres are compared. The dyeing problems in relation to the latter are discussed and it is stated that there is a need for future new fibres to possess polar groups in order to give better dyeing properties.

A. H.

Newer Fibres—Some Dyehouse Problems. I. M. S. Walls. *Dyer*, 110, 909-911 (11 Dec. 1953).

Dyeing problems in connection with polyamide, polyester, polyacrylic, and polyvinyl fibres are described, and the use of "dye-carriers" and high-temperature dyeing methods as means of solving some of the problems are discussed.

A. H.

PATENTS

Dyeing Textiles. Rydboholms. *BP* 704,972

The material is impregnated with the dye liquor, heated in one single layer to fix the dye, rolled up on itself and the roll rotated while maintaining the temperature constant and the vapour pressure of the liquor in the material in equilibrium with the vapour pressure of the surrounding atmosphere, rotation being continued long enough to ensure uniform distribution with fixation of the dye in the material.

C. O. C.

Improved Exhaustion of Metachrome Dyes. Ciba. *BP* 705,879

Exhaustion of metallisable wool dyes when dyeing by the single-bath metallising process, e.g. in metachrome dyeing, is increased by presence in the dyebath of a non-resinous water-soluble reaction product of ≤ 4 mol. of an $\alpha\beta$ -alkylene oxide with 1 mol. of an organic compound containing at least one basic primary or secondary amino group or a basic tertiary amino group together with an alcoholic OH group and contains ≥ 2 N atoms bound to the same C atom. This compound may be present either in the free state or as a salt, sulphuric ester or a quaternary ammonium compound. Thus 1% of the dye 1-hydroxy-2-amino-4-nitrobenzene \rightarrow 1,3-diaminobenzene-4-sulphonic acid applied by the normal metachrome method yields a very weak dyeing, addition of 1% of the reaction product of 6 mol. ethylene oxide and 1 mol. dodecylamine results in a strong brown dyeing being produced.

C. O. C.

Vat Dyeing of Cellulose Acetate. Lansil. *BP* 705,332

Addition of small amounts of pyridine or crude pyridine bases to an aqueous mixture of vat dyes (except those containing a single anthraquinone nucleus linked at two adjacent C atoms to a second cyclic residue through a heterocyclic grouping) and hydrosulphite or hydrosulphite-based reducing agents produces great increase in the rate of reduction of the dye even when the amount of the base is such that the pH of the vat remains at < 7 . Vats made in this manner dye cellulose acetate without the use of the swelling agent.

C. O. C.

Phosphorescent Yarns. J. Goldstein. *USP* 2,635,969

Yarns of all fabrics, but particularly nylon yarns, are rendered phosphorescent by treating them with activated crystals of metallic salts such as ZnS. The crystals should pass through a 90-325 mesh screen and are suspended in a vehicle or lacquer in which to obtain the best results the ratio of plasticizer to resin used is 8-20:1. The handle and other properties, except for the phosphorescence, of the treated yarns retain their original character.

C. O. C.

Dyeing Chrome Leather. Ciba. *BP* 705,335

Improved dyeings are obtained on chrome or combination tanned leather if as a wetting agent there is used before or during dyeing the reaction product of ≤ 4 mol. of an $\alpha\beta$ -alkylene oxide, e.g. ethylene oxide, with 1 mol. of an organic compound containing at least one basic, primary or secondary amino group or containing a basic tertiary amino group and in addition an alcoholic OH group, e.g. oleylamine.

C. O. C.

Gas-fading Inhibitors. General Aniline. *BP* 703,500

NN'' -Dibenzyl- $NN''N''$ -trialkyldialkylenetriamines either alone or mixed with symmetrical dialkyl dibenzyl alkylene diamines and symmetrical, $NN''N''$ -dibenzyl- $NN''N''N''$ -tetraalkyltrialkylenetetramines are excellent gas-fading inhibitors for use in the dyebath.

C. O. C.

Chemistry of Chelation in Textile Processing (III p. 194).

Colouring Materials by Producing Iron Hydroxide Within Them (V p. 204).

Addition Reaction of Phenyl isoCyanate with Bleached Ramie (VI p. 205).

Cross-linking in Cellulose Fibres. I—Physical and Mechanical Properties of Cellulose Fibres treated with Diisocyanate (VI p. 206).

Influence of Various Classes of Dyes on the Strength of Nylon on Exposure to Light (VI p. 206).

Removal of Water from Textiles (X p. 210).

IX—PRINTING

Photochemistry of Dyes in Solution. G. Oster. *Phot. Eng.*, 4, 173-8 (1953); *Chem. Abs.*, 48, 475 (25 Jan. 1954).

A review of photobleaching of dyes in solution. Binary dye combinations in which one dye acts as a photosensitiser are proposed as practical bleach-out systems, e.g. a mixture of Acriflavine and Crystal Violet.

C. O. C.

PATENTS

Printing Screens. J. Hlubucek. *BP* 704,148

The impermeable parts of the screen are coated with a polyvinyl chloride composition which is then heat cured yielding a screen of high resistance to acids, alkalies, solvents, salts, and aqueous liquids without the need for a protective lacquer.

C. O. C.

Screen Printing with Resinous or Thermoplastic Colours. R. Welsh. *BP* 703,177

When applying resinous or thermoplastic colours, e.g. to metal or ceramics, a metal screen is used which is heated by passing an electric current through it, or alternatively a silk screen is used with an overlaying series of wires immediately adjacent to it through which electric current is passed. The screen is brought to a temperature at which the colour is sufficiently molten to be forced through the screen by a squeegee.

C. O. C.

Multicolour Printing. John Dickinson & Co. *BP* 704,775

A method of obtaining very accurate registration when printing two or more colours upon transparent material, e.g. regenerated cellulose film in which one colour is applied to one side of the film and another colour at the other side.

C. O. C.

Decorated Coating of Thermoplastic Synthetic Resinous Material. J. H. L. Galuska and A. J. Mejicek. *BP* 705,684

A monochrome layer of a thermoplastic resin, preferably polyvinylchloride, is applied to a carrier sheet of cloth or paper. A pattern is then printed on the coating with a thermoplastic synthetic resin dispersed in a non-solvent non-volatile plasticiser, this composition having a temperature-softening curve approximating to that of the coating. The coating and the applied pattern are then fused together by heat.

C. O. C.

Azo Dye Duplicating Process. A. B. Dick Co. *USP* 2,634,677

The image is formed on the master, e.g. of gelatin, preferably in reverse, of a water or insoluble component of a water insoluble dye and which has no objectionable colour value in the state in which it is applied to the master. Upon transfer to the copy sheet this component reacts with other dye forming components contained in a non-reactive state in the master or in the fluid with which the copy sheet is wetted to effect transfer of the image or in the copy sheet or in a fluid applied to the copy sheet after the image has been transferred.

C. O. C.

Multi-coloured Photographic Prints. A. Mayer and E. Schoenborn-Buchheim. *BP* 702,903

An image is developed in an exposed silver halide emulsion on a support either by treatment with a tanning developer, or with an ordinary developer followed by a hardening bleaching agent and then redevelopment, to form a relief image. This relief image is transferred to an intermediate support and the soluble halide emulsion washed out with warm water. The silver retained in the relief image is converted into a coloured substance either by toning or by converting the silver into a mordant, e.g. silver thiocyanate, and dyeing it. The image or images are then transferred in register on to paper or other permanent base.

C. O. C.

Colour Photography. B. Gaspar. *BP* 705,171

Azo dyes are used which at one pH are in the transmittent form and at another pH in an absorbent form. This enables cyan dyes to be transformed so that they have little absorption in the red end of the spectrum during exposure but can be returned to the normal colour after developing.

C. O. C.

Screen Printing (I p. 193).

Investigations on Colour Development. IX—Behaviour of Sulphite-containing Colour Developers towards Colloid-free Silver Salts in Presence of a Dye-forming Component. (IV p. 199).

Azoic Coupling Components: Relationship between Chemical Structure and Substantivity for Cellulose. (VIII p. 208).

X—SIZING AND FINISHING

Removal of Water from Textiles. H. Rath. *Textil-Praxis*, 9, 65-68 (Jan. 1954).

A fundamental examination of the amount of water removed from textiles by mechanical treatments such as squeezing, hydroextraction, and suction is described, including the influence of variations in temp. of treatment and pressure of the squeeze rollers. In padding dye solns. the expression of water should be kept low in order to produce full colours, but this varies with the degree of absorption of the individual dye.

B. K.

Tear Strength of Resin-treated Textiles. A.A.T.C.C. Piedmont Section. *Amer. Dyestuff Rep.*, 43, P 41-P 45 (18 Jan. 1954).

One cotton and two rayon fabrics are treated with various amounts of urea-formaldehyde resin and sulphur, and tested for tensile and tear strengths using a wide variety of methods. Results indicate that certain tear tests provide a better indication of quality than tests more commonly used in industry. An impact tear test is best for resin-treated fabrics, and both rayon and cotton cloths lose tear strength as the resin content increases, cotton being most affected. Softeners are shown to improve tear strength.

J. W. B.

PATENTS

Slip Resistant Finish. Monsanto. *USP* 2,635,056

Impregnating yarn or fabric with a silica aquasol containing 1-20% by weight of a water soluble polyhydric alcohol so as to deposit 0.5-5.0% silica in the fibres imparts an excellent slip resistant finish together with a soft, full handle and with no tendency to dust off.

C. O. C.

Crease-Resisting Finish. K. Quehl. *BP* 703,432

The material is impregnated with an aqueous solution of formaldehyde and a co-ordination complex of urea whose cation has the formula $(Me(\text{CON}_2\text{H}_2)_n)_n$ (Me = metal of valency > 1 ; n = a whole number) and then dried at $< 100^\circ\text{C}$. This imparts an excellent crease resisting finish which is fast to washing. No baking is needed and the liquor used is very stable.

C. O. C.

Woven Multi-ply Narrow Fabrics. Thomas French & Sons. *BP* 705,481

Fabric of greater accuracy of gauge than is obtainable from the loom, better appearance and with a controlled degree of stiffness hitherto impossible is obtained by impregnating, if desired after piece dyeing, a multi-ply narrow fabric of the kind having at least one main web with interwoven formations at predetermined pitch or spacing, with an aqueous soln. of a thermosetting resin and drying so as to set the resin.

C. O. C.

Resin Finish. Tootal Broadhurst Lee Co. *BP* 704,143

Material wholly or partly of regenerated cellulose is impregnated with a urea-formaldehyde precondensate and an acid catalyst and then cured by heating in an atmosphere containing $< 20\%$ of superheated steam. The finished material is much more resistant to abrasion than if the curing is effected in absence of superheated steam.

C. O. C.

Flame-resistant Finish. Federal Leather Co. *USP* 2,634,218

The material is impregnated with an aq. soln. of SbF_3 and dried. It is then heated with an aq. carbonate or bicarbonate of an alkali metal or ammonia, rinsed and dried.

C. O. C.

Water-repellent and Mildew-resistant Finishes by Using Organosilicon Compounds. Cowles Chemical Co. *BP* 705,260

An insoluble organosilicon compound is deposited in the material from an aqueous solution at $\text{pH} > 7$ of the products of the alkaline hydrolysis of an organo-trihalogenosilane, after which the impregnated material is dried and baked to complete polymerisation of the silicone resin. The solution used is made preferably by the method

of *BP* 675,188 and 675,233. The effect is enhanced if, after impregnation and drying but before baking, the material is treated with an aqueous solution of the salt of an amphoteric material.

C. O. C.

Reducing the Felting Power of Wool. Ciba. *BP* 704,896

Modification of *BP* 675,137 (*J.S.D.C.*, 68, 417 (1952)). The wool is wet-chlorinated in presence of a water-soluble alkyl ether of dimethylolurea, especially the dimethyl or diethyl ether of dimethylolurea, this addition helping to give more uniform chlorination.

C. O. C.

Coating Hard Fibre Fabric with Rubber. H. Boinet and E. Boinet. *BP* 703,718

A hard fibre (e.g. hemp) fabric is treated with a rubber composition, and then without intermediate vulcanisation, coated on a calender with more rubber, and finally vulcanised. There is not the loss in strength that is experienced when rubber is calender coated onto hard fibre fabric without preliminary treatment with a rubber composition.

C. O. C.

Nylon Crêpe Fabric and Yarn. A. L. Dargin. *BP* 705,274

Nylon and silk are twisted together in a proportion of 75:50:25:50 by wt. The yarn may then be either woven up or crimped. The fabric or yarn is next boiled off in the normal manner when shrinkage of the silk imparts a crêpe effect. The material is then heat set to fix the nylon and finally the silk is removed with caustic alkali and the material neutralised. This caustic treatment may be given before heat setting as the boiling off gives sufficient set to the nylon to allow for the removal of the silk after such setting.

C. O. C.

Treating Socks, etc. to confer Immunity Against Athlete's Foot and similar Infections. Fairforest Co. *USP* 2,633,446

The goods are treated for 5 min. in a 1–10% (preferably 8%), aqueous solution of the lauryl pyridinium salt of 5-chloro-2-mercaptopbenzothiazole, dried and baked for 5 min. at 230°F. This gives protection against infection with e.g. *Trichophyton interdigitale*.

C. O. C.

Desizing of Glass Fibre Fabrics and their subsequent Coating. Alexander H. Kerr & Co. *USP* 2,633,428

The size is burnt from the fabric which is then treated with silicone which results in the finished fabric having an exceptionally full handle. Machinery is described in which both processes are carried out continuously and simultaneously. The preferred silicone is a 50% emulsion of methyl silicone, curing being carried out at 450–700°F. This treatment also makes the fabric water-repellent, this property being unimpaired by subsequent washing and/or hypochlorite bleaching.

C. O. C.

Treating Glass Fibres with Organosilicon Compounds. Cowles Chemical Co. *BP* 705,261

Glass fibres are rendered water-repellent and their resistance to abrasion enhanced by treating them with an aqueous solution at pH > 7 of the products of the alkaline hydrolysis of an alkyl trichlorosilane (Alk of 4–18%), drying and baking.

C. O. C.

Glyoxal and its Derivatives in the Textile Industry (III p. 194).

Mothproofing Agents for Wool. XIII—Relation between the Chemical Constitution of Mothproofing Agents and their Effects on Wool (III p. 194).

Production of Pile Fabrics by Electrostatic Deposition (VI p. 207).

Removing Mineral Oil from Vegetable Fibres with Simultaneous Flameproofing of the Fibres (VII p. 207).

Effect of Urea-Formaldehyde Resins on the Light Fastness of Direct Cotton Dyes (VIII p. 208).

Fastness Properties of Acetate Rayon Dyeings after-treated with Curable Resins (VIII p. 209).

Decorated Coating of Thermoplastic Synthetic Resinous Material (IX p. 210).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Accessible Cellulose as measured by Sorption of Sulphuric Acid. G. A. Richter, L. E. Herde, and I. L. Gage. *Ind. Eng. Chem.*, 45, 2773–2779 (Dec. 1953).

Sorption of H_2SO_4 from acetic acid by water-swollen cellulose depends not only on the sorption conditions, but on the history of the cellulose. For most celluloses, there is a linear relationship between H_2SO_4 sorption and water vapour sorption at 95% R.H., indicating that measurement of the former is a means of estimating a type of cellulose accessibility. The best relationship is found when the *M* values (max. H_2SO_4 sorption from conc. soln. in acetic acid, calculated by extrapolation of the isotherms) are plotted against the water vapour sorption, a straight line passing through or near the origin being obtained. Similar plotting of the sorption of H_2SO_4 from dil. soln. in acetic acid by a number of sulphite pulps and ligner-base celluloses gave a straight line which intersected the water vapour sorption axis, and unmercerised Kraft pulps showed sorptions too low to fall on this line. These results are considered to show that the capacity of the water-accessible fractions for sorption of H_2SO_4 can vary between celluloses, indicating fundamental differences in the properties of the "accessible" fractions of celluloses of different histories. With increasing severity of mercerisation, the higher accessibilities measured by vapour sorption methods are paralleled by H_2SO_4 sorption.

W. K. R.

Fractional Separation of Cellulose—Theoretical Considerations. R. L. Scott. *Ind. Eng. Chem.*, 45, 2532–2537 (Nov. 1953).

From a theoretical study of the solution thermodynamics of high polymers, as applied to cellulose, it is concluded that—it is unnecessary to subdivide into more than a few fractions per stage; fractionation is sharper from more dilute solutions, but careful refractionation is better than working at dilutions much < 1 g./litre, and a combined precipitation and solution method is superior to either alone. Insufficiently sharp fractionation may cause serious errors in mol. wt. distributions and in the viscosity–mol. wt. relationship. Other methods of fractionation, e.g. chromatography, would be expected to give better results if a satisfactory method could be developed.

W. K. R.

Summative Cupriethylenediamine Fractionation of Cellulose. W. A. Mueller and L. N. Rogers. *Ind. Eng. Chem.*, 45, 2522–2526 (Nov. 1953).

Cellulose may be fractionated by selective extraction with dilute solutions of cupriethylenediamine. A method is described in which only data on the concentrations of cellulose and the viscosities of the extracts are required to give a summative distribution curve, which then may be expanded mathematically to provide integral and differential distribution curves. Results compare favourably with distribution curves obtained by several different methods on standard samples.

W. K. R.

Recent Work on the Molecular Weight of Cellulose. E. H. Immergut, B. G. Rånby, and H. F. Mark. *Ind. Eng. Chem.*, 45, 2483–2490 (Nov. 1953).

A relationship has been established between the degree of polymerisation of cellulose, as determined by measurement of the osmotic pressure of the nitrate in acetone, and the intrinsic viscosity of the primary cellulose measured in cupriethylenediamine. Attempts to measure the osmotic pressure of cellulose in cupriethylenediamine, using polyvinylbutyral and polytrifluorochloroethylene membranes, met with some success. The shear dependence of the intrinsic viscosity of cellulose nitrates in acetone was studied, and it is concluded that above mol. wt. of 300,000 the influence of the shear gradient becomes considerable.

W. K. R.

Intrinsic Viscosity of Cellulose: Report of the Cellulose Disperse Viscosity Subcommittee. A. F. Martin. *Ind. Eng. Chem.*, 45, 2497–2499 (Nov. 1953).

A summary is presented of work being carried out to produce a standard method for determining the intrinsic viscosity of cellulose. The results of a study of solvent

power and stability of cupriethylenediamine and cuprammonium solvents as functions of the Cu and base content, are described.

W. K. R.

Intrinsic Viscosities and Molecular Weights of Cellulose and Cellulose Derivatives. E. H. Immergut and F. R. Eirich. *Ind. Eng. Chem.*, **45**, 2500–2511 (Nov. 1953).

The hydrodynamic theory of high polymer solutions shows that there is a relationship between molecular dimension, rather than molecular weight and viscosity. Molecular models which connect weight with dimensions have to be adopted, and the effect of these on viscosity can be calculated. When the viscosities of solutions of cellulose, extrapolated to zero concentration and shear, are compared for different molecular weights, it is found that up to an average degree of polymerisation (D.P.) of approx. 200 the relation for rods is obeyed. Thereafter is a transition until D.P. 400–600, when the equations for coiled models are obeyed. Above D.P. 1000 impenetrable coils are indicated. With cellulose nitrate and acetate rather higher degrees of polymerisation are required to obtain the same states of flexing than for cellulose in copper solvents. The usual good solvents modify the viscosities by further stiffening the chains as solvent power increases, but these effects are smaller than those due to the nature of the substitution.

W. K. R.

Degree of Order in Fine Structure of Regenerated Cellulose Films. A. I. Zaïdes and I. G. Stoyanova-Sinitkaya. *Doklady Akad. Nauk S.S.R.*, **92**, 601–602 (21 Sept. 1953).

Electron-diffraction measurements are made on cast film formed by regeneration of cotton cellulose dissolved in cuprammonium soln. Previous X-ray-diffraction work showing the presence of microcrystalline regions in such film is confirmed, and it is shown that the values of the spacings obtained by the two methods are identical.

A. E. S.

Preparation of Cellulose Xanthates of Maximum Degree of Xanthation. E. Geiger and B. J. Weiss. *Helv. Chim. Acta*, **36**, 2009–2017 (Dec. 1953).

The preparation of cellulose trixanthate is described, and it is shown that xanthation in solution depends on two equilibrium reactions, viz. formation of alkoxide (ONA) groups on the one hand and their interaction with CS₂ on the other. In accord with the law of mass action, when the NaOH and CS₂ conc. are increased, trixanthates may be obtained. Two analytical methods are described for the determination of the γ -number at high degrees of xanthation.

H. H. H.

Stable Derivatives of Cellulose Xanthate. I. Aleksandru and Z. Rogovin. *J. Gen. Chem. U.S.S.R.*, **23**, 1199–1203 (July 1953).

Stability properties of cellulose derivatives that may be conventionally represented as Cell-O-CS-SH (I), Cell-O-CS-S-CH₃-COOH (II), Cell-O-CS-NH₂ (III), and Cell-O-CS-S-CH₃ (IV) are examined. II, III, and IV are very much more stable than I to hydrolysis by water, acids, or alkalies, to heat, and to storage under various conditions, being comparable in these respects with cellulose acetate. IV is prepared from the Na salt of I (viscose) by treatment with (CH₃)₂SO₄, and it is shown that no change in degree of esterification of the cellulose occurs during the reaction; the Na salt of I treated with sodium chloroacetate yields the Na salt of II, from which II itself is readily obtained; and the Na salt of II reacts with ammonia to give III.

A. E. S.

Distribution during Xanthation of Xanthic Ester Groups between the Primary and Secondary Alcohol Groups of Cellulose. I. Aleksandru and Z. Rogovin. *J. Gen. Chem. U.S.S.R.*, **23**, 1203–1205 (July 1953).

Viscose, as normally prepared, and a product of its further xanthation are treated with (CH₃)₂SO₄ to stabilise the ester groups (see previous abstract). They are then treated with triphenylmethyl chloride (which etherifies only the primary alcohol groups), and analysis of the products enables the distribution of xanthic ester groups between primary and secondary alcohol groups to be determined (ratio found = 1 : 4).

A. E. S.

Solvent Power of Solvents for Cellulose Acetate.

W. R. Moore, J. Russell, and J. A. Epstein. *Chem. and Ind.*, 1339–1340 (12 Dec. 1953).

Osmotic pressures have been determined for soln. of secondary cellulose acetate (53.7% acetic acid yield) previously used in viscosity and initial phase separation studies, the solvents used being acetone, methyl acetate, nitromethane, pyridine, α -, β - and γ -picolines, dioxan, and acetic acid. The Huggins interaction const. μ (\propto solvent power) were derived from the expression—

$$\frac{\pi}{c} = \frac{RT}{M_p} + \frac{RTd_s(\frac{1}{c} - \mu)}{M_sd_p^2}$$

(c = polymer concn., π = osmotic pressure, d = density, and M = mol. wt.; p and s refer to polymer and solvent respectively). The results, which are tabulated, show that there is no apparent correlation between μ and any suggested measure of solvent power derived from viscosity. For the cellulose acetate studied, and within the range of solvents used, the vol. of non-solvent required to cause initial phase separation (the dilution ratio) appears, however, to give an approx. measure of solvent power in a thermodynamic sense; the diln. ratio and μ are mutually inversely variable. The soln. in dioxan alone gave anomalous results. The experimental procedures are given in some detail.

J. W. D.

Unit Cell and Preliminary Structure for Cellulose Triacetate.

W. J. Dulmage. *Nature*, **172**, 1053 (5 Dec. 1953).

Small films of highly crystalline triacetate with good fibrous orientation and a fair degree of biaxial orientation are prepared by repeated rolling at successively higher temp. between 160 and 215°C. The higher orientation is improved by swelling in glacial acetic acid, and then drying at 110°C. under pressure. X-Ray diffraction photographs show several new spacings, and enable all spots to be indexed on the basis of an orthogonal unit cell containing four cellobiose hexa-acetate residues. Failure to set up a model which will give certain reflexions, however, leads to the conclusion that cellulose triacetate is only pseudo-orthorhombic.

J. W. B.

Intrinsic Viscosity of Cellulose Nitrate related to Degree of Nitration.

C. H. Lindaley and M. B. Frank. *Ind. Eng. Chem.*, **45**, 2491–2497 (Nov. 1953).

The degree of nitration of cellulose nitrate of a given molecular weight has an important effect on the viscosity of its solutions. An empirical equation has been derived whereby the intrinsic viscosity of the trinitrate may be calculated from the experimentally determined viscosity and N content of any cellulose nitrate. Thus it is possible to compare the intrinsic viscosity and hence the degree of polymerisation of cellulose nitrate containing different amounts of N. Values for the Staudinger constant have been recalculated to allow for the effect of variable degree of nitration.

W. K. R.

Assignment of Structure to Cellulose 3:6-Dinitrate.

E. L. Falconer and C. B. Purves. 5th Canadian High Polymer Forum. *Chem. and Ind.*, 13 (2 Jan. 1954).

Segall and Purves (*Canadian J. Chem.*, **30**, 860 (1952)) showed that a soln. of NH₂OH in pyridine eliminated ca. one NO₂ group from cellulose trinitrate, but they were unable to determine the structure of the resulting compound. Their methylated cellulose dinitrate has now been successfully reduced to a methyl cellulose of 1 : 1 substitution with NH₂ hydroxylphide. When hydrolysed with acid, this methyl cellulose gave an 81% yield of partly methylated sugars, of which 84% was isolated as cryst. 2-methyl glucose by chromatography on a cellulose column. The remainder was 2:3- and perhaps 2:6-dimethyl glucose. The original denitration was probably restricted to NO₂ groups in the second positions of the cellulose trinitrate.

J. W. D.

Decomposition of Lignin by Metallic Sodium in Liquid Ammonia. VI—Action on Fir Wood of Sodium in Liquid Ammonia.

A. F. Semechkin and N. N. Shorýgina. *J. Gen. Chem. U.S.S.R.*, **23**, 1593–1597 (Sept. 1953).

Fir wood treated below –33°C. in a soln. of Na in liquid NH₃ is broken down, yielding polysaccharides (cellulose and hemicelluloses) containing some methyl groups (indicating that not all the methoxyl content of wood is to be attributed to the presence of lignin) and some

unchanged lignin and its decomposition products, from which dihydroxygenol, obtained previously from "isolated" lignin (see *J.S.D.C.*, **69**, 475 (Nov. 1953)), is isolated. It is concluded that native and isolated lignin are essentially similar in nature. A. E. S.

PATENTS

Hectographic Carbon Papers. H. A. O'Neill.

BP 705,167

Paper is coated first with a hectograph transfer coating and then with a continuous non-tacky protective film consisting of wax dissolved or dispersed in a hydrocarbon or halogenated hydrocarbon solvent, neither wax nor solvent dissolving the dye in the hectograph coating. The hectograph coating comprises a spirit-soluble dye in waxes, oils and plasticiser, and is applied at about 95°C. The coating and the protective layer may be applied in one pass through a carbon machine provided with a second coating roller with associated trough and scraper for the protective coating. S. V. S.

Coated Paper suitable for Stylus Inscription. Nashua Gummed & Coated Paper Co. BP 705,417

A record sheet suitable for receiving inscriptions from a stylus comprises a supporting layer of sheet material, e.g. paper, and an obscuring film covering its surface consisting of thermoplastic resinous material with a three-dimensional cellular structure formed by uniformly distributed microscopic or submicroscopic voids that render the film opaque. The pressure of the stylus causes the voids to collapse locally, releasing air and resulting in a homogeneous transparent area being formed. The resinous material may be ethyl cellulose (with a compatible resin to harden the film, if desired), polystyrene resin, polyvinyl resin, a copolymer of vinyl chloride and vinyl acetate, polymethyl methacrylate or a cellulose ester. The obscuring film is applied as an emulsion of a volatile liquid, e.g. water, with an added substance to reduce its vapour pressure, in the form of multitudinous droplets, in the solution of the thermoplastic, film-forming, resinous material, the film dried, to gel the resinous material, and the volatile droplets evaporated to give the cellular structure. S. V. S.

Coating Paper. Robert Gair Co. USP 2,632,714

The paper is first treated with an insolubilising agent and while still damp is coated with an aqueous pigment suspension containing an adhesive or with an aqueous solution of an adhesive. On heating in the short drying space between the coater and finishing calender the coating is insolubilised yielding a product that may be subjected to the action of numerous solutions applied at the waterbox on the finishing calender. C. O. C.

Glossing Paper (I p. 193).

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Structure of Surface-denatured Protein. V—Surface Films of Diketopiperazine. K. Imahori. *Bull. Chem. Soc. Japan*, **26**, 439–441 (Nov. 1953).

Measurements of surface pressure, potential, and viscosity of diketopiperazine films spread on 0.2N-KCl soln., and plotting those values against surface area, all give a limiting area for diketopiperazine of 34.6 sq. Å. per molecule. By comparison with X-ray diffraction data (Corey, *J. Amer. Chem. Soc.*, **60**, 1598 (1938)) it is concluded that diketopiperazine molecules lie flat on the surface. The limiting area per amino acid residue of diketopiperazine (17.3 sq. Å.) is much greater than the mean limiting area per amino acid residue in a natural protein (e.g. 12.5 sq. Å. for horse serum albumin) and in synthetic polypeptides (14.7 sq. Å. for polyglycine). It is concluded that surface layers of polypeptides do not have the diketopiperazine configuration. A. J.

Surface Chemistry of Synthetic Protein Analogues.

II—Temperature Dependence of Force-Area

Relations of some Polypeptide Monolayers and Thickness of the Monolayers. J. Isomura and K. Hamaguchi. *Bull. Chem. Soc. Japan*, **26**, 425–429 (Nov. 1953).

Force-area curves for surface films of poly-DL- α -aminolauric acid, poly-DL- γ -aminodecanoic acid, poly-DL- α -amino-octanoic acid, and poly- α -benzyl-DL-glutamate all show a horizontal section parallel to the A

axis, a decrease in area occurring at constant surface pressure. The horizontal section occurs at lower pressure values as the temp. increases. The limiting area before the pressure again increases is in each case 15 sq. Å. per amino acid residue. With poly-DL- α -aminohexanoic acid, the copolymer of glycine and alanine, and natural proteins, no horizontal section is observed. It is concluded that the decrease in area at constant pressure is due to re-orientation of the side-chains where these contain a chain of more than four atoms. A. J.

Thermal Contraction of Collagen and its Dissolution with Elastase. I. Banga, D. A. Hall, R. E. Tunbridge, and G. C. Wood. *Nature*, **172**, 1099–1100 (12 Dec. 1953).

It is suggested by the first author that collagen fibrils are surrounded by a mucopolysaccharide-like envelope bound by dicarboxylic acids; when this is removed by heat denaturation, elastase digests it rapidly, suggesting that in both collagen and elastin certain identical structural elements are present. The remaining authors demonstrate that elastase will attack collagenous material even when it has not been thermally contracted, and also disagree with some of the first author's conclusions. J. W. B.

Soluble Reconstituted Collagen. D. S. Jackson and J. Ball. *Nature*, **172**, 1046 (5 Dec. 1953).

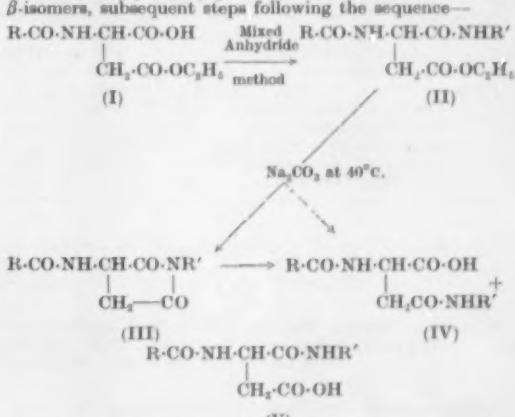
Fibrous precipitates, obtained by adding sodium chloride to solutions of animal tendon in dilute acetic acid, are prepared, dissolved, and reformed under varying conditions, and a mechanism is suggested for the building up of fibre aggregates. J. W. B.

α and β -Forms of Poly-L-alanine. C. H. Bamford, L. Brown, A. Elliott, W. E. Hanby, and I. F. Trotter. *Nature*, **173**, 27–29 (2 Jan. 1954).

Poly-L-alanine is prepared in both α - and β -forms, the X-ray diffraction pictures of which are discussed and interpreted. A remarkable similarity is reported between the β photograph and a diffraction photograph of Tussah silk. J. W. B.

Specific Fission of Peptide Links by Chemical Means. A. R. Battersby and J. C. Robinson. *Chem. and Ind.*, **45** (9 Jan. 1954).

Normal α -aspartyl peptides are first converted to the β -isomers, subsequent steps following the sequence—



Realisation of the scheme (I) with R = C₆H₅, R' = H, and (2) R = C₆H₅, R' = CH₂-CO-NH-C₆H₁₅ is described fully; in the first instance the step from (II) to (IV) + (V) passes via (III). There is evidence that the method is of wide application. J. W. D.

PATENTS

Tanning, Dyeing and Finishing Fish and Reptile Skins. H. Rose. USP 2,633,730

The skins are fleshed, descaled etc. in the normal manner and then, if desired after being bleached, are tanned with a vegetable or synthetic tanning agent. After fat-liquoring they are dyed at 100°F. with an acid dye. They are then folded flesh side inwards around a pipe through which cold water is flowing and while on this pipe they are

singed to burn off the tips of the scale pockets using a flame or plate at 800°r. Finally they are brushed, glazed and plated, preferably under a Sheridan plating machine at 200°r. A very smooth finish and a two tone effect is achieved.

C. O. C.

Water-resistant Leather by Use of Aminosilane Resins. N. D. Cheronis and E. L. Gustus.

USP 2,635,059

Leather or other porous material is rendered water-resistant by treating it with a solution or dispersion of an organic solvent solution of a precondensate of an aminosilane resin together with modifiers, plasticisers and extenders. The solvent is dried off and polymerisation completed. The treated leather remains permeable unless excess of resin over that required for the water-resistant finish is used.

C. O. C.

Water-resistant Leather. J. Greenebaum Tanning Co.

USP 2,635,060

The damp leather, previously completely or partially tanned, is treated with an aqueous dispersion of an organic solvent solution of certain resins, plasticisers, binding agents and emulsifying agents. The resins used are preferably made from silicon-containing compounds.

C. O. C.

Protein Structure (VI p. 206).

Removing Hair and Wool from Hides and Skins (VI p. 207).

Flame-resistant Finish (X p. 210).

XIII—RUBBER; RESINS; PLASTICS

Dye-sensitised Photopolymerisation. G. Oster. *Nature* 173, 300–301 (13 Feb. 1954).

Acrylonitrile (7%) in aqueous solution buffered at pH 7, with fluorescein or a halogenated derivative as a sensitiser, is readily polymerised on irradiation with visible light, in presence of oxygen and, in the solution, a mild reducing agent, ascorbic acid and phenylhydrazine hydrochloride being particularly effective. It appears that in presence of air the reduced dye DH, reacts with atmospheric O₂ to give the semiquinone form DH and hydroxyl radicals—



The free radicals thus produced initiate the polymerisation of the monomer. Certain other monomers can also be similarly polymerised. The choice of reducing agent to be used in conjunction with a given dye is critical. In general, the reduction potential of the dye, at a given pH, should be more negative than that of the reducing agent, so that the dye is not decolorised in the dark. The reduction potential of the dye should also be within about 2 electron-volts of that of the reducing agents, this being a necessary condition for the photoreduction of the dye by visible light. Oxygen concentration is also critical. Certain monomers, e.g. acrylamide, are themselves reducing agents and may readily be photosynthesised without added reducing agents.

J. W. B.

Viscosities of Extremely Dilute Polystyrene Solutions in Toluene. D. J. Streeter and R. F. Boyer. 5th Canadian High Polymer Forum. *Chem. and Ind.*, 14 (2 Jan. 1954).

Microcalorimetric Studies of Polyvinyl Acetate and Polystyrene Solutions. M. Rinfret. 5th Canadian High Polymer Forum. *Chem. and Ind.*, 14 (2 Jan. 1954).

Application of Theoretical Dilute Solution Viscosity Equations to Commercial High Copolymers. R. B. MacFarlane and L. A. McLeod. *Chem. and Ind.*, 12 (2 Jan. 1954).

Synthesis of Branched Methyl Polymethacrylate. G. Smets and J. Schmetz. *Bull. Soc. Chim. Belg.*, 62, 358–371 (July–Sept. 1953).

The copolymerisation of methyl methacrylate and small amounts of ethylidene dimethacrylate enables branched methyl polymethacrylate to be obtained. A kinetic study of the reaction now permits the rate of ramification to be expressed as a function of the degree of progress of the reaction and of a const. *k*, which is equal to the ratio of the const. of velocity of propagation and of ramification of the polymeric chains. In this particular case *k* = 0.7.

H. H. H.

Catalytic Action of Organic Peroxides on the Polymerisation of Ethenoid Compounds. II—Activity of Substituted Benzoyl Peroxides on the Polymerisation of Vinyl Acetate. M. Takebayashi, T. Shingaki, and Y. Ito. *Bull. Chem. Soc. Japan*, 26, 475–479 (Nov. 1953).

The effect of *pp'*-dimethoxy-, *mm'*-dimethoxy-, *pp'*-dimethyl-, *mm'*-dimethyl-, unsubstituted, *pp'*-dichloro-, *mm'*-dichloro-, *pp'*-dicyano-, *mm'*-dicyano-, *pp'*-dinitro-, and *mm'*-dinitro-benzoyl peroxides on the anaerobic polymerisation of vinyl acetate has been measured at 40° and 60°C. The peroxides form two groups, *pp'*-dimethoxy- to *pp'*-dichloro-benzoyl peroxides inclusive having high efficiency, the remainder having low efficiency. In both groups the overall velocity of polymerisation, measured at a steady state, is proportional to the square root of the dissociation constant of the corresponding substituted benzoic acid. The decomposition of the peroxides measured iodometrically, is of the first order at 40°C., when CO₂ is not evolved except from *pp'*-dimethoxybenzoyl peroxide. At 60°C. decomposition is not of the first order, and CO₂ is evolved from the first group, when free aryl radicals are assumed to be formed. It is concluded that the rate of polymerisation is a function of both the rate of decomposition of the peroxides and also the activity of the radicals produced.

A. J.

Solvent Power of Various Solvents for High Polymers. J. Kawai. *Bull. Chem. Soc. Japan*, 26, 409–413 (Nov. 1953).

The use of the Huggins parameter μ as a measure of thermodynamic interaction between a polymer and solvents, and as a measure of solvent power, is criticised on the grounds that the size and the shape of the unit segment of the polymer chain and of the solvent molecule may not be identical. The effects of segment size on osmotic pressure, viscosity, and swelling are discussed, and an expression—

$$\frac{1}{V_1} - \frac{\mu_g + \Delta\mu}{V_1}$$

(V_1 = metal volume of solvent molecule) is derived, which gives better agreement with the observed volume swelling ratio for solutions of divinylbenzene gels in 26 solvents than does μ .

A. J.

Surface Chemistry of High Polymers. I—Non-electrolytic Flexible Linear Polymers at Air-Water Interface. T. Isemura, H. Hotta, and T. Miwa. *Bull. Chem. Soc. Japan*, 26, 380–385 (Sept. 1953).

Surface films of polyvinyl acetate (P.V.A.), polyvinyl stearate (P.V.S.), and copolymers, spread on water from benzene soln., and of varying surface concn., have been examined and relations between surface pressure, surface potential, and the area occupied per monomer unit obtained. Polyvinyl acetate gives an expanded film and the co-area per monomer is about twice the area per monomer calculated from the molecular model. With polyvinyl stearate, a compressed film is obtained, the observed co-area being equal to the calculated area per monomer. With copolymers, compressed films are again obtained, and the area per monomer is often less than that for P.V.S. alone. It is concluded that with P.V.A. the acetate side-chains lie in the interface and molecular motion is possible; whilst with P.V.S. the stearate chains stick out above the surface and associate with each other. With copolymers, association of stearate chains may force acetate chains into the water phase.

A. J.

Surface Chemistry of High Polymers. II—Non-electrolytic Flexible Linear Polymers at Oil-Water Interface. H. Hotta. *Bull. Chem. Soc. Japan*, 26, 396–399 (Sept. 1953).

Interfacial pressure–surface area (π –*A*) relations for monolayers of amilan (ω -aminohexanoic acid polymer), polyvinyl acetate, polyvinyl stearate, and vinyl acetate–stearate copolymers have been obtained at a petroleum ether (b.p. 100–140°C.)–water interface. The π –*A* curves for the vinyl polymers are coincident for values of *A* > 50 sq. Å. per vinyl unit, but with decreasing *A* (increasing π) deviation occurs in descending order of stearate content. This is in contrast to the behaviour at an air–water interface. The π –*A* curves for amilan show reversals at ca. 18 and 38 sq. Å. These are interpreted as a transition from the flat-in-surface to the bent-into-oil forms of the hydrocarbon part of the main chain, and the dimensions

are consistent with the molecular models. It is concluded that polymer films at the oil-water interface are expanded except at low values of A , and that normally there is no mutual interaction of stearate side-chains in the oil phase.

A. J.

Alkali Compounds of Polyvinyl Alcohol. S. N. Ushakov and E. M. Lavrent'eva. *J. Appl. Chem. U.S.S.R.*, **26**, 960-968 (Sept. 1953).

A polyvinyl alcohol preparation is treated with NaOH solutions, and the amount of NaOH absorbed by the polymer, determined from the changes in concn. that occur in the soln., is found to increase as the concn. of the soln. increases, reaching a max. (10 g. of NaOH absorbed by 100 g. of polymer from a 24% soln. of NaOH at 20°C.), and then falling again to zero (at 37% NaOH). The non-absorption of NaOH from solutions of high concn. is considered to be related to the fact that these solutions, unlike those of lower concn., do not wet the surface of the polymer, and it is attributed to the restriction of the reaction to the surface under these conditions. Changes in temp. (0-60°C.) and changes in the physical form of the polymer (including those brought about by its reprecipitation from aq. soln. by gradual addition of NaOH soln.) have little effect on the results, and the polymer does not appear to suffer degradation in any of the treatments given. The alkali cpd. of polyvinyl alcohol formed in this way and the Na deriv. formed by treatment of the polymer with Na in liquid NH₃ are found to have identical infrared absorption spectra, and the alkali cpd. is therefore considered to be of the alkoxide type.

A. E. S.

Chemical and Physical Properties of Alkyl Aryl Phosphates. H. R. Gamrath, R. E. Hatton, and W. E. Weesner. *Ind. Eng. Chem.*, **46**, 208-212 (Jan. 1954).

The preparation and significant chemical and physical properties of several alkyl diaryl phosphates are described. The esters are used as plasticisers, particularly for polyvinyl chloride and its copolymers. Their properties are intermediate between those of trialkyl and triaryl phosphates.

W. K. R.

Epoxy Fatty-acid Ester Plasticisers. F. P. Greenspan and R. J. Gall. *Ind. Eng. Chem.*, **45**, 2722-2726 (Dec. 1953).

The introduction of an epoxy group, by reaction with peracetic acid, into the fatty-acid chain of esters of e.g. oleic, soya-bean, and cottonseed fatty acids greatly improves their compatibility with polyvinyl chloride (P.V.C.) resins. In general, the epoxy fatty-acid esters were equal to or better than dioctyl phthalate as plasticisers, and some showed excellent low-temperature flexibility. Permanency varied from fair to excellent according to structure and mol. wt. P.V.C. formulated with these esters possessed outstanding stability to heat and light, even in the absence of added stabilisers.

W. K. R.

PATENTS

Stabilised Polyester Compositions. Libbey-Owens-Ford Glass Co. *USP* 2,632,751

Nuclearly substituted arylhydroxides having at least one nuclear substituent that (a) consists at least in part of a monovalent hydrocarbon radical and (b) has no O atom connected to an atom that is connected to the nucleus, the total number of O and N atoms that are connected to any atom that is connected to a nucleus being < 2 , e.g. guaiacol, stabilise a mixture of a polymerisable unsaturated polyester and a polymerisation catalyst against polymerisation at atmospheric temperatures, i.e. polymerisation does not occur until the mixture is heated.

USP 2,632,752

A material which upon polymerisation has improved water resistance and electrical properties consists of dehydrated kaolin and a polymerisable unsaturated polyhydroxy alcohol-polycarboxylic acid polyester containing several polymerisably reactive $\Delta^{1,5}$ -enediyl groups.

USP 2,632,753

p-Benzoquinone is an excellent stabiliser for mixtures of polymerisable unsaturated polyesters and polymerisation catalysts against polymerisation at room temperature.

C. O. C.

Fungi- and Bacteria-resistant Polyvinyl Halide Compositions. Monsanto. *USP* 2,632,746

Copper 8-quinolinolate is rendered compatible with plasticised polyvinyl halide compositions by incorporation of a small amount of cadmium or calcium ricinoleate. The copper 8-quinolinolate neither crystallises nor blooms under any conditions of application or use of such compositions.

USP 2,632,747

Copper 8-quinolinolate is rendered compatible with plasticised polyvinyl halide compositions by incorporation of an *N*-alkyl arylsulphonamide but if such compositions are not kept exposed to ultra violet radiation then after some time the copper 8-quinolinolate begins to bloom. This can be prevented by adding cadmium or calcium ricinoleate.

C. O. C.

Index of Chemicals in Reports and Documents on the German Chemical Industry. Part I—Index to Published Reports. Part II—Index to Unpublished Reports and Documents (III p. 193).

Colouring Materials by Producing Iron Hydroxide Within Them (V p. 204).

Rubber Degradation Products suitable as Coating Materials (V p. 204).

Plastic and Textile Materials (VI p. 207).

XIV—ANALYSIS; TESTING; APPARATUS

Anomalies in Colorimetric pH Measurements. M. L. Macon. *Teintex*, **18**, 622-645 (Oct. 1953).

Indicators are unsatisfactory for pH measurements of soln. containing surface-active compounds, only pH meters with glass electrodes being satisfactory. The introduction of surface-active cpd. into dyebaths may exert an appreciable effect on the pH values.

B. K.

German Standard Methods for Auxiliary Products—Assessment of Products for Prevention of Lime Soap Formation. DIN 53,903. *Textil-Praxis*, **8**, 1063 (Dec. 1953).

A standard method is described for assessing the value of auxiliary products for the prevention of lime soap precipitation, employing sodium oleate in water of 40° German hardness (34° calcium and 6° magnesium salts).

B. K.

Spectrophotometric Determination of Glycerol as Sodium-Cupri-Glycerol Complex. F. Spagnolo. *Anal. Chem.*, **25**, 1566-1568 (Oct. 1953).

The procedure due to Whyte (*Oil and Soap*, **23**, 323 (1946)) is modified to enable smaller quantities of glycerol to be determined, and the range of application of the procedure to be widened. Sensitivity is increased by the use of absorption cells of longer light path, and titration of the glycerol soln. to a faintly perceptible turbidity is eliminated by the use of a definite amount of reagent and a working aliquot of soln. to contain 17-66 mg. of glycerol for the colour development. The method is applicable to glycerol esters and resinous vehicles. Optimum stability of the coloured complex obtains within the range 400-1800% excess of reagent ensured by the method.

J. W. D.

Chromatographic Separation of Methyl Glucoses.

W. J. Whelan and K. Morgan. *Chem. and Ind.*, **78**, (16 Jan. 1954).

The method of Whistler and Durso (*J. Amer. Chem. Soc.*, **72**, 677 (1950)) may be used to resolve mixtures of partly methylated glucoses. As the number of methoxyl groups per molecule increases, so also does the concn. of ethanol required to effect elution. Data are reported for the resolution of an artificial mixture of glucose, 2:3-di-, 2:3:6-tri-, and 2:3:4:6-tetra-*O*-methyl glucoses, each component being recovered in 93-100% yield. The difference in degree of adsorption on charcoal between tri- and tetra-methyl glucoses is much greater than between di- and tri-methyl glucoses. Experimental procedure is described.

J. W. D.

Paper Chromatography of some Carbohydrates and Related Compounds in the presence of Boric Acid. G. R. Barker and D. C. C. Smith. *Chem. and Ind.*, **19-20** (2 Jan. 1954).

Formation of ionised complexes of polyhydroxy compounds with boric acid depends on stereochemical factors.

R_f values are recorded which were obtained by running chromatograms on paper impregnated with boric acid and developing with *n*-butanol saturated with boric acid-saturated aq. boric acid; they are not physical constants but relative values which are reproducible if the experimental conditions are closely adhered to. Certain mixtures of methylated sugars may be resolved by this method, and where separation is normally achieved, presence of boric acid enhances the degree of resolution. Sugars incapable of complex formation have unchanged R_f values, e.g. fully methylated sugars. The steric aspect of complex formation with methylated sugars is discussed at some length. The above technique facilitates also the separation of some acyclic polyols, but is less successful with unsubstituted sugars. Complex formation and consequent change in R_f value are closely paralleled by the enhancement of the conductivity of boric acid soln. when the same carbohydrates are present. J. W. D.

Production of Formaldehyde during the Oxidation of Carbohydrates with Periodate. J. F. O'Dea. *Chem. and Ind.*, 1338-1339 (12 Dec. 1953).

The two mol. of HCHO theoretically expected from the periodate oxidation of maltose and lactose are never obtained. Oxidations carried out at 50°C. suggest that two reactions are involved in which HCHO is, respectively, produced and recombined; the final shape of the HCHO-time curve at various temp. depends on the rates of these reactions. The HCHO (max. value) derived by oxidation at room temp. and pH 3 gives an accurate estimate of chain length, being produced by the oxidation of the glycolic-like structure on C_4 and C_6 of the non-reducing end-group of the chain. It is suggested (and there is supporting evidence for this) that HCHO recombines principally with "active hydrogen" structures, carboniums being formed which readily combine with HCHO; these proposed end-group structures are shown. J. W. D.

Polarographic Determination of Nitrite. D. Tung-Wei Chow and R. J. Robinson. *Anal. Chem.*, 25, 1493-1496 (Oct. 1953).

Nitrite gives a current enhancement, starting at zero applied potential, which is different from that of nitrate, and which depends on nitrite concn. The molybdate-catalysed reduction of NO_3^- in both SO_4^{2-} and Cl^- soln. and the reduction of NO_3^- in Cl^- soln. have been studied, and show that the current enhancement due to both NO_3^- and NO_2^- is greater in Cl^- soln. Low concn. of NO_3^- may be determined, and a method is proposed for the determination of NO_3^- and NO_2^- in presence of each other. Full details are given, and typical polarograms are shown and discussed. J. W. D.

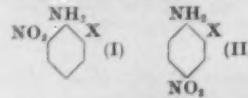
Determination of Ammonium, Amide, Nitrite, and Nitrate Nitrogen in Plant Extracts. J. E. Varner, W. A. Bulen, S. Vanecko, and R. C. Burrell. *Anal. Chem.*, 25, 1528-1529 (Oct. 1953).

The extract, freed from proteins, is buffered at pH 10 with borate, placed in a modified semi-micro-Kjeldahl distillation unit, and the ammonium N removed by vacuum distillation at 40°C. After addition of conc. alkali the amide N is removed by steam distillation at 100°C. Ferrous sulphate is added as a reducing agent, and the nitrite N is removed as NH_3 . Finally the nitrate N is reduced to NH_3 by $FeSO_4$ after addition of Ag_2SO_4 as catalyst. The entire procedure, which is fully detailed, requires ca. 20 min. Interferences (due to glucose and to certain amino acids) are discussed together with methods for overcoming them. There is a sketch of the apparatus. J. W. D.

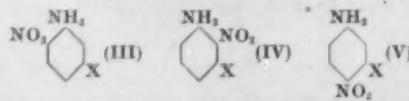
Quantitative Chromatographic Separation of *ortho*- and *para*-Nitroanilines and their Monosubstituted Derivatives. J. E. Larson and S. H. Harvey. *Chem. and Ind.*, 45-46 (9 Jan. 1954).

The quant. resolution of *o*- and *p*-nitroanilines may readily be effected on an activated alumina column, the chromatogram being subsequently developed and eluted with benzene containing varying proportions of ethyl acetate, vis. 20% ethyl acetate for development and for elution of the *o*-isomer (lower band), and 40% for elution of the *p*-isomer (upper band); when the *m*-isomer is present it forms a band midway between the other two. The quant.

results of three such separations are given. The same technique has been used to separate subst. nitroanilines—



where $X = NO_2, Cl, Br$, I order of absorption (I) \ll (II)



where $X = NO_2, F, Cl, Br$, I order of absorption (III) \ll (IV) \ll (V)

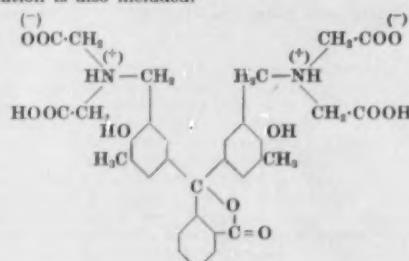
Where $X = CH_3$ or $COOC_2H_5$, (III) and (IV), though not resolved, were easily separated from (V). J. W. D.

Fluorometric Estimation of Naphthols. D. N. Vaskovich. *J. Appl. Chem. U.S.S.R.*, 26, 1213-1217 (Nov. 1953).

To 1- or 2-naphthol as a soln. in 50% alcohol (1.5 ml.), 0.1 N-NaOH (0.5 ml.) is added just before fluorometric examination (visual comparison with standards). The limit for detection is 0.2 μ g. of naphthol, and that for quantitative estimation is 0.5 μ g. A. E. S.

Metal Indicators. VII—A Phthalein binding Alkaline Earth Ions and its Analytical Application. G. Anderegg, H. Flaschka, R. Sallmann, and G. Schwarzenbach. *Helv. Chim. Acta*, 39, 113-120 (Jan. 1954).

A deriv. (I) of phenolphthalein, carrying 2 iminodiacetic acid groups, which enable the compound to bind metal cations, is described. Complex formation with Mg^{2+} , Ca^{2+} , Sr^{2+} , Ba^{2+} , and Zn^{2+} is investigated quant., and the absorption spectra of the different complexes are also given. The new substance, called phthalein-complexone, is an excellent indicator for the complexometric titration of alkaline earth metals. A method for sulphate determination is also included.



H. H. H.

Determination of Ascorbic Acid by a New Colorimetric Method. M. Schmall, C. W. Pifer, and E. G. Wollish. *Anal. Chem.*, 25, 1486-1490 (Oct. 1953).

The determination is based upon the reaction of ascorbic acid with diazotised 4-methoxy-2-nitroaniline in acid soln., followed by development of a blue colour, whose max. absorbance is at 570 μ m, in alk. soln. The mechanism is not yet known. The method is detailed and fully discussed; interferences are indicated. J. W. D.

Thin-film Interferometry as an Aid in the Examination of Fibre Topography. A. Skortchly. *J. Textile Inst.*, 45, T 78 (Jan. 1954).

Fibres having a topography which is normally difficult to observe, e.g. fine cashmere, are cleaned in benzene and light petroleum, dipped in a solution of 1 part by vol. of Canada balsam and 50 parts of benzene, and dried in warm air near a Bunsen flame. They are then mounted longitudinally on a microscope slide, being held in position by strips of adhesive tape at the ends. On being viewed in reflected monochromatic light (sodium vapour tube) using an objective $\times 0.66$, magnification $44\times$, a definition of cuticular structure as good as with a phase-contrast microscope is obtained. J. W. B.

Fibre Identification. H. Sieber. *Dyer*, 110, 901-907 (11 Dec. 1953).

Orthodox methods (microscopic, Shirleystain, and Neocarmine) are described; in addition, the Zillo test set, consisting of a range of selective solvents and solvent combinations specially chosen for rapid identification of the newer fibres, is discussed. A. H.

Flexible Fibre-bundles for Transporting Optical Images. A. C. S. van Heel. *Nature*, 173, 39 (2 Jan. 1954).

Glass or, preferably, plastic fibres are coated with either plastic of low refractive index or other transparent material of low refractive index, and made up into bundles, a thin coating of black paint first being applied to isolate each fibre from its neighbours. Suitable refractive indices are 1.52 for the core and 1.47 for the coating. Images formed on one end of the bundle are effectively transmitted to the other end, even with the bundle bent through 360°. J. W. B.

Flexible Fibrescope using Static Scanning. H. H. Hopkinson and N. S. Kapany. *Nature*, 173, 39-41 (2 Jan. 1954).

The transmission of light along the inside of monofils by internal reflexion is discussed theoretically, and a method is described of constructing a bundle of well aligned glass fibres of 0.0025 in. diameter. A 4 in. length, even when bent to an arc of 2 in. radius, transmits sharp images from one end to the other. J. W. B.

Determination of Small Amounts of Chromium in Textiles by means of Diphenylcarbazide. A. G. Hamlin. *J. Textile Inst.*, 45, T 44-T 54 (Jan. 1954).

Textile material containing Cr is dry-ashed if no interfering ions are present, and otherwise is destroyed by wet combustion with nitric, sulphuric, and perchloric acids. Removal of traces of Fe and Cu is essential, and is done as their oxime complexes, from a solution of suitable pH value, by means of chloroform. Details are given of interference by other ions, and procedures described for removing Sn, Ti, Zr, Sb, and Hg should these also be present. The acidity and salt concentration of the solution are then adjusted, and the Cr is oxidised to the hexavalent state by boiling with ammonium ceric sulphate, excess ceric ion being reduced with hydrazoic acid before determination of Cr. Oxidation with ammonium ceric sulphate is found superior to that with hypobromite for determination of soluble Cr in cloths treated with hydrated Cr oxides. The intensity of the colour given by different batches of diphenylcarbazide with identical Cr solutions is also found to vary greatly. J. W. B.

Molecular Weight Uniformity of Cellulose and Cellulose Derivatives—Recent Developments in Measurement. C. M. Conrad. *Ind. Eng. Chem.*, 45, 2511-2517 (Nov. 1953).

A critical review of recent developments in methods for measuring molecular weight uniformity. Fractionation methods are the most common, and precipitation fractionation is considered to be superior to solution fractionation. New methods discussed include measurements based on chromatographic adsorption, ultracentrifuge sedimentation, diffusion, turbidity, flow birefringence, and electron micrometry. W. K. R.

Precipitation Fractionation of Cellulose Nitrate. R. L. Mitchell. *Ind. Eng. Chem.*, 45, 2526-2531 (Nov. 1953).

A tentative A.C.S. (American Chemical Society) standard method is presented for determining the molecular weight distribution of cellulose. The method which is based on the precipitation fractionation of the cellulose nitrate derivative, involves standardised procedures for (1) conversion of the cellulose into cellulose nitrate, (2) separation into fractions by stepwise precipitation from solution by addition of a non-solvent, and (3) measurement of the weight and viscosity of each fraction and calculation of its degree of polymerisation. W. K. R.

Determination of Lignin in Partly Delignified Jute. W. G. Macmillan, A. B. Sen Gupta, and A. Roy. *J. Textile Inst.*, 45, T 108-T 112 (Feb. 1954).

Samples of partly delignified jute are prepared from dewaxed jute of 13.97% lignin content, using the sodium chlorite method. Their lignin contents are determined by

treating 1.0-1.5 g. of material with 72% H_2SO_4 , using 12.5 ml./g. of sample, for different times at 2°C., and then diluting the mixture to 3% concn. and refluxing for 2 hr. The amount of condensation product formed during the isolation of lignin is obtained from the difference of the values of lignin corresponding to 0 and 48 hr. of treatment. The effects of varying the original amount of acid, and of varying the duration of refluxing the diluted solution, on the yield of lignin are also studied. Some of the isolated lignin samples are analysed for non-lignous matter, and the average value is determined. Losses in lignin content due to the different methods are calculated by the extrapolation procedure discussed earlier in the paper. The correct lignin content so obtained is compared with the usual 72% H_2SO_4 at 2°C. method, and with the value from the method using strong acid at 22°C. for 1 hr., and is shown to be closely similar. It appears, therefore, that the two latter procedures, which entail no correction factors and are thus simpler, can safely be adopted for partly delignified jute. J. W. B.

Estimation of Fibrolane BX or Viscose Rayon in Blends with Wool. A. N. Davidson and R. Preston. *J. Textile Inst.*, 45, T 142-T 146 (Feb. 1954).

Mixtures of wool and Fibrolane, BX (approx. 1 g. samples) are boiled for 6 min. in 100 ml. of a fresh solution prepared by stirring 5 g. of hydrated $Ba(OH)_2$ and 20 g. of KNO_3 in 100 ml. of boiling distilled water until dissolved. The residue is filtered hot through a sintered glass crucible (Pyrex SF 2B1), washed successively with 60 ml. of hot 2N-HCl, 200 ml. of hot water, and 10 ml. of acetone, and finally dried to constant wt. at 100-105°C. Wool is preferentially soluble, and although 20% of the Fibrolane BX is also dissolved, the variability between different experiments is low, and suggests that the fraction soluble in $Ba(OH)_2-KNO_3$ soln. is constant. For blends containing 10% or more of Fibrolane BX the equation—

$$y = 1.4 x - 11$$

may be used (y = actual % and x = % found, of Fibrolane BX). The method is applied to yarns and tops and data are supplied. The procedure is also applied to known viscose rayon-wool blends, when it is found that, by making allowance for an insoluble residue (2.5%) obtained by treating wool alone, exceedingly good agreement is reached. J. W. B.

Relaxation of Stretched Animal Fibres. I—Apparatus for Determining Relaxation Curves. G. C. Wood and N. H. Chamberlain. *J. Textile Inst.*, 45, T 147-T 161 (Feb. 1954).

An apparatus is described for measuring the rate of decay of tension which occurs after wool and other animal fibres have been rapidly stretched by a fixed amount. Earlier apparatus, depending on the observation of a stretched spring, was useless for measuring the decay in the very brief initial stages. The new device utilises a measurement of the deflection, due to a tension applied perpendicular to it, of a flat steel beam clamped at both ends. The beam is previously calibrated, and the extremely small displacements involved are determined by means of a parallel-plate condenser, one plate of which is fixed and the other attached to the beam. The change in capacity of this condenser is converted to a D.C. voltage change which is recorded oscillographically.

Rapid extension of the fibre is effected by a strong coiled spring in conjunction with an oil-damped piston, the damping being variable, thus permitting variation in the rate of extension. Release of the spring triggers the oscilloscope, and a trace is obtained which records a continuous measure of tension from the commencement of extension to some 240 msec. later. Long-term relaxation is measured by a beam-and-spring device incorporated in the same apparatus, the measurement being by eye and travelling microscope. Although there is a time-lag of some 30 sec. between the two measurements, the points can be easily joined up on load-log time graph. Interpretation of the curve is not discussed at this stage. J. W. B.

Acid Solubility Estimations for determining Modifications of Wool by Chemical Treatments. H. Zahn and A. Würz. *Textil-Praxis*, 8, 971-974 (Nov. 1953).

Alkali solubility estimations represent the standard method of determining modifications in wool by chemical

treatments. Methods of using acids as an alternative have been studied, and hydrochloric acid has been found to give satisfactory results based on various chemically modified wools from bulk processing. The wool is treated in 4 N-HCl for 60 min. at 65°C. B. K.

Catalytic Activity of Cysteine and Related Compounds in the Iodine-Azide Reaction. D. W. Whitman and R. McL. Whitney. *Anal. Chem.*, **25**, 1523-1527 (Oct. 1953).

The catalytic activity of cysteine and related compounds in the evolution of N_2 by the iodine-azide reaction has been measured in Warburg respirometers at pH 4-63; evidence was obtained as to the mechanism of the reaction, and this is discussed at length. The vol. of N_2 evolved is quant. related to the thiols and disulphide present and is also dependent upon the time of reaction, the temp., the reagent concn., and the group attached to the thiol group. The method is not, without further study, suitable for the measurement of mixtures of thiol groups, but its specificity may be of value in following changes in available thiol groups in complex biological systems. J. W. D.

Specific Test for Hydroxyproline in Paper—Chromatographic Separation of Amino Acids. J. B. Jepson and I. Smith. *Nature*, **172**, 1100-1101 (12 Dec. 1953).

Proposed International Tests for Colour Fastness of Textiles. International Organisation for Standardisation ISO/TC 38/SC 1. J.S.D.C., **69**, 409-429 (Nov. 1953); *Amer. Dyestuff Rep.*, **42**, P871-P887 (21 Dec. 1953).

General principles of colour fastness testing are described and details given of the grey scales for evaluating change in colour, and staining. This is followed by 25 tests, comprising—spotting with acid and alkali, hypochlorite and peroxide bleaching, $AlCl_3$ and H_2SO_4 carbonising, acid chlorination, daylight, mercerising, Cr salts in dye-bath, Fe and Cu salts in dye-bath, organic solvents, potting, hot pressing, rubbing, soda boiling, stoving, hand washing, water and sea water by the flat dish, test tube, and Perspirometer methods, and water spotting. J. W. B.

Construction and Use of a Semi-micro Light-scattering Apparatus. D. A. I. Goring. *Canadian J. Chem.*, **31**, 1078-1092 (Nov. 1953).

Details are given for the construction of a light-scattering apparatus requiring only 4 ml. of liquid, which enables the measurement of intensity for angles between 37.5 and 135° to the incident beam. In this range the unpolarised, horizontal, and vertical components of the light emitted from a solution of fluorescein are const. to within $\pm 0.2\%$, 0.4%, and 0.6% respectively. H. H. H.

Influence of the Refractive Index on the Absorption of Light by Solutions. J. Schuyer. *Rec. Trav. chim.*, **72**, 933-949 (Nov. 1953).

Starting from the theory of Onsager and Böttcher (*J. Amer. Chem. Soc.*, **58**, 1486 (1936); *Physica*, **9**, 939, 945 (1942)) a new formula is derived whereby the influence of the refractive index on the mol. extinction coefficient of a solution can be calc. Suitable approximation formulas are given for certain types of solutions which are in better agreement with experiment (e.g. the refractive indices of 9,9'-difluorene in cyclohexane, CCl_4 , and C_6H_6) than the formula of Chako and Kortum (*J. Chem. Phys.*, **2**, 644 (1934); *Z. physikal. Chem.*, **B 33**, 243 (1936)). H. H. H.

Colour Adaptation and Colour-discrimination Thresholds. G. N. Rautian. *Doklady Akad. Nauk S.S.R.*, **92**, 297-299 (11 Sept. 1953).

In an anomaloscope, one half-field is maintained constant in colour and intensity and the other is varied in such a way that stimulus changes occur in only one set of receptors in the observer's eye. A normal observer views the field continuously for 10 min., and his colour-discrimination threshold is determined at intervals. The experiment may be preceded by a 5-min. adaptation of the eye in the dark, or there may be no form of controlled adaptation [the extent to which the type of adaptation used affected the results is not made clear]. For every normal observer and for each set of receptors, power of discrimination is found to increase rapidly after viewing has commenced, the discrimination threshold being approximately halved in 3 min., when a fairly steady state is attained. Only

after 10 min. are there signs, in some cases, of reduced sensitivity due to fatigue. The test is applied to a partial protanope, when it gives normal results for the green and blue receptors; for the red receptors, however, the threshold is initially six times the normal value and rises to 10 times the normal value in 10 min. (a condition of virtual dichromatism). A. E. S.

Colour-rendering Properties of Fluorescent Lamps.

B. H. Crawford. *Nature*, **173**, 100-102 (16 Jan. 1954).

A survey of a symposium organised by the Colour Group of the Physical Society. J. W. B.

Sixth Annual Review of Analytical Chemistry. *Anal. Chem.*, **26**, 2-181 (Jan. 1954).

The 25 headings include—X-ray diffraction, electron microscopy, chemical microscopy, organic polarography, acid-base titrations in non-aqueous media, chromatography, ion exchange, and extraction. There are copious references to the literature. J. W. D.

Estimation of Water Content of Small Amounts of Proteinaceous Material. H. Sobel. *Anal. Chem.*, **25**, 1756 (Nov. 1953).

Material is brought into contact under specified conditions with Karl Fischer reagent which has been diluted with anhyd. methanol, and the colour is measured spectrophotometrically. The result is used in conjunction with a prepared standard curve. The diluted reagent is stable for several days if suitably stored. A complete determination requires <10 min. J. W. D.

Determination of Moisture in Salts by the High-frequency Method. F. W. Jensen and M. B. Burton. *Regional Conclave of the American Chemical Society: Anal. Chem.*, **25**, 1932 (Dec. 1953).

Apparatus consists of a 10-megacycle oscillator with a sample column placed in series with a diode rectifier and a D.C. microammeter; a temp. compensator is provided. Samples (50 g.) of salt mixtures are agitated for 5 min. with a mixture of 1:4 dioxan-methanol; after centrifuging, the liquid is introduced into the sample column and readings are taken on the microammeter. The solvent is always saturated with salt and its conductance rises with increase in moisture content; with the correct concn. of dioxan-methanol the response is linear. Good accuracy, reproducibility, time stability, and temp. compensation are claimed. J. W. D.

Determination of Water in Alcohols by an Indirect Conductivity Method. C. K. Hancock, C. M. Hudgins, and R. E. Zerwekh. *Regional Conclave of the American Chemical Society: Anal. Chem.*, **25**, 1932 (Dec. 1953).

Studies of methyl or ethyl alcohol-acetone-water saturated NaCl systems show that conductivity is nearly linear with water content over the range 0-10% water. Linearity is more closely approached by increasing the acetone concn., but there is a parallel decrease in sensitivity; optimum acetone concn. is $\sim 30\%$. Application of the method to the determination of water in other materials is of considerable interest, and good results have been obtained in the determination of water in soils and in sucrose soln. J. W. D.

Copper-Ethylenediaminetetra-acetic Acid Complex in Alkaline Solution—Qualitative Comparisons with Benedict's Reagent. *Anal. Chem.*, **25**, 1925-1926 (Dec. 1953).

Copper-ethylenediaminetetra-acetate reagent prepared with Na_2CO_3 at pH 10.5 is more sensitive than the complex containing $NaOH$ at the same pH for the qual. determination of glucose in aq. soln., but is less sensitive than Benedict's reagent in the qual. estimation of glucose in urine. Substantiating experimental evidence is presented and discussed. J. W. D.

Simultaneous Titration of Iron and Copper with Ethylenediaminetetra-acetic Acid—Spectrophotometric End-points. A. L. Underwood. *Anal. Chem.*, **25**, 1910-1912 (Dec. 1953).

The Cu^{2+} complex absorbs strongly at $745\text{ m}\mu$, at which wavelength the Fe^{2+} complex has no absorption. Both complexes have large stability constants, and since the Fe^{2+} complex is the more stable, formation of the Cu^{2+} complex, with resulting increase in absorbance, serves as indicator for the titration of the Fe^{2+} ; the Cu^{2+} end-point is subsequently shown by attainment of a steady max.

value for absorbance. If present, Al may be substantially pptd. as the chloride, the small remainder being insufficient to interfere. The method is fully described. J. W. D.

Determination of Primary Fatty Amines in Amine Mixtures—Potentiometric Titration Techniques. J. E. Jackson. *Anal. Chem.*, 25, 1764–1765 (Nov. 1953).

By substituting CHCl_3 for CH_3OH as solvent, the method of Wagner, Brown, and Peters (*J. Amer. Chem. Soc.*, 69, 2611 (1947)) may be applied to distinguish primary from secondary and tertiary amino groups in long-chain fatty amines; it is particularly effective when primary and secondary amino groups are present in the same mol. The procedure is described and the results of trial analyses quoted. The amines must be protected against atmospheric CO_2 and, if present, carbonates must be decomposed. Aqueous isopropanol is added to the CHCl_3 soln. to improve the form of the titration curve and to enhance the accuracy of very small secondary amine titrations. J. W. D.

Titration of Weak Bases in Acetic Anhydride—Solvent Mixtures. J. S. Fritz and M. O. Fulda. *Anal. Chem.*, 25, 1837–1839 (Dec. 1953).

Addition of excess (5%) acetic anhydride removes the last traces of water from soln. of bases in org. solvents; when most tertiary amines and alkali-metal salts are titrated in such media with HClO_4 , this considerably increases the sharpness of the break and rise in potential at the end-point. Very large excess of acetic anhydride does not further improve the sharpness of the end-point. The method, which is given in full, gives excellent results for tertiary amines including nitrogen heterocyclics of the purine, pyridine, pyridone, and thiazole types, but primary, secondary, and a few heterocyclic amines cannot be titrated. Potentiometric titration curves are shown for a number of amines and for lithium nitrate. Visual indicators are also valuable, and Methyl Violet, 1-naphtholbenzoin, Neutral Red, triphenylmethanol, and dibenzalacetone have all been found to be effective. J. W. D.

1-Fluoro-2:4-dinitrobenzene as a Quantitative Reagent for Primary and Secondary Amines. F. C. McIntire, L. M. Clements, and M. Sproul. *Anal. Chem.*, 25, 1757–1758 (Nov. 1953).

The reagent is caused to react with the amine at 60°C. in alkaline soln. After dilution with dist. water the reaction mixture is extracted with cyclohexane or, in the cases of ethanolamine or other highly water-soluble amines, *s*-tetrachloroethane. The absorbance of the solvent extract is measured at a wavelength indicated by a table given in the text. The method, even if not superior to others in current use, should be of value in the determination of primary and secondary amines in presence of tertiary amines, and it is applicable to highly water-soluble amines. Certain amines are prevented by steric hindrance from reacting. Quantities of the order of 0.1 micromole of an amine may be determined. J. W. D.

Photometric Titration in Non-aqueous Solvents. C. N. Reiley and B. Schweizer. Regional Conclave of the American Chemical Society: *Anal. Chem.*, 25, 1934 (Dec. 1953).

In a study of the application of the photometric method to acid-base titrations in non-aqueous media, weak bases were titrated in acetic acid soln. with HClO_4 in the same solvent. The titration cell was placed directly in the light path of a spectrophotometer and the absorbance at an experimentally determined wavelength was plotted against the corrected vol. of titrant. Bases such as *o*-chloroaniline, *m*-chloroaniline, and quinoline gave distinct breaks at the point of equivalence, agreeing well with potentiometric end-points. Where neither acid nor base forms show absorbance in the ultraviolet region, successful titrations were carried out by addition of an absorbing species of weaker basicity; successive end-point breaks indicate the vol. equivalent to the quantity of each component. J. W. D.

Determination of Schiff Bases by Titration in Non-aqueous Solutions. S. K. Freeman. *Anal. Chem.*, 25, 1750–1751 (Nov. 1953).

Schiff bases (ten have been studied) may be successfully titrated with HClO_4 in acetic acid soln., to a potentiometric

end-point, or Methyl Violet may be used as a visual indicator. Owing to the levelling influence of the acetic acid, this method will not differentiate between a Schiff base and its parent amine, but this may be accomplished if CHCl_3 or acetonitrile is used as solvent. Differentiation proved impossible by these procedures in two instances. Typical titration curves, and the results of trial analyses are given. J. W. D.

Effects of Time and Temperature on the Reaction of Formaldehyde with a Modified Schiff's Reagent. R. Hatfield and E. C. Smith. Regional Conclave of the American Chemical Society: *Anal. Chem.*, 25, 1934 (Dec. 1953).

The Schiff-Deniges method has been studied over a range of conditions of 0–25 hr., at temp. of 20°, 25°, and 30°C., with varying concn. of HCHO . A colour development time of 10 min. at 25°C. was established as a standard for routine analysis. Experiments with both basic fuchsin and rosaniline hydrochloride showed that the form of the dye produced differences in colour intensities; one brand of rosaniline hydrochloride was selected as standard. When the dichromate ion was present in concn. < 100 p.p.m., a change of wavelength from the standard 580 μm . to 650 μm . gave results in which all values were within 10% of the mean. Acetals derived from HCHO are completely hydrolysed when 1 ml. H_2SO_4 is added to 5 ml. acetal soln. and allowed to stand 15 min. at ca. 65°C., the resulting HCHO can then be determined by the above method; the test may be used qual. or quant. with respect to HCHO . J. W. D.

Polarographic Analysis of Lead Driers. D. A. Skoog and R. L. Focht. *Anal. Chem.*, 25, 1922–1924 (Dec. 1953).

Most of the common lead driers (except lead resinate) readily disperse in aq. dodecylamine acetate (I) to clear soln. These dispersions give typical polarographic waves for Pb, the wave heights being directly related to the concn. of Pb in the driers. The half-wave potentials for the different soaps vary only slightly, and are not materially different from that of Pb acetate. The concn. of (I) is important. A recommended procedure is detailed which is rapid, simple, and free from the time-consuming and tedious operations associated with existing (gravimetric and titrimetric) methods. Lead resinate is imperfectly dispersed, and yields low results. J. W. D.

Determination of the Non-volatile Content of Paint Media. Oil and Colour Chemists Association. *J. Oil and Col. Chem. Assocn.*, 36, 649–653 (Nov. 1953).

The known non-volatile contents of five media have been determined by an oven-heating procedure employing two variants each of time (3 and 4 hr.) and temp. (110°C. and 130°C.). Nearly 1,000 results have been submitted by 24 operators in different laboratories; these have been analysed statistically in order to determine (1) the accuracy (variation from the true known value), and (2) the precision (reproducibility) of the methods. The best of the methods examined, for accuracy and precision, is heating for 4 hr. at 110°C. The largest error is that between operators. The results are detailed. J. W. D.

Can We Measure the Light Fastness of Pigments? G. A. Campbell. *J. Oil and Col. Chem. Assocn.*, 36, 646–648 (Nov. 1953).

The present system for assessing the light fastness of a pigment in that of referring its fading performance to that of a "Standard Madder Lake" in terms of a numerical scale whose max. value is 10. No attempt is made to specify the form in which the pigment is to be exposed, or the conditions of exposure. The excellent work of the S.D.C. in evolving the Blue Scale, and in recognising that the light fastness of a dye cannot be determined—only that of a dyeing—is acknowledged. A co-operative investigation is to be organised by the Light Fastness Committee of the O.C.C.A., three stages being visualised—(1) To give members an opportunity of using the B.S.1006 for themselves. This will allot an index number to a pigmented coating or a pigmentation. (2) By the accumulation of these data, and perhaps by a defined and accepted method of incorporating a pigment into a medium, devise a secondary index number to be

attached to a *pigment* itself as distinct from pigmentations. This would be a derived figure and would give general indications only, but it would serve a useful purpose in industry when discussing pigments in general terms. (3) Longer-term work involving a study of the instrumentation and the mechanism of fading.

J. W. D.

PATENTS

Humidity Meter. A. Vogt.

BP 704,552

A humidity meter operating by variation in electrical conductivity of an indicator substance mounted between two electrodes uses as the indicator substance an inorganic substance difficultly soluble in water and of thickness $< 10^{-3}$ mm. measured perpendicularly to the gap between the electrodes, e.g. a metal fluoride or an inconvertible metal.

C. O. C.

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A tube has three vertical limbs leading to closed vessels and is partly filled with liquid. One vessel is kept wet by a wick supplied with water, another is kept dry at the ambient temperature, both these vessels containing air or other gas. The third vessel is kept dry at the ambient temperature and contains a substance which does not mix with the liquid in the tube and is different from the gas in the other two vessels. When the liquid in the tube reaches a given level in the limb leading to the wet vessel it energises an electric relay which sets in motion operating, indicating and control means.

C. O. C.

Apparatus for Decanting Tests. Sulzer Frères.

BP 703,132

A container has a removable cover and is divided horizontally into two compartments by a partition wall having perforated portions forming a chamber in which samples to be tested are placed, e.g. two perforated discs between which the sample is placed the discs being forced together when the cover is fixed in position. Steam is then blown through the container under controlled conditions.

C. O. C.

Tear Strength of Resin-treated Textiles (X p. 210).**Intrinsic Viscosity of Cellulose: Report of the Cellulose Disperse Viscosity Subcommittee** (XI p. 211).**Preparation of Cellulose Xanthates of Maximum Degree of Xanthation** (XI p. 212).**Solvent Power of Solvents for Cellulose Acetate** (XI p. 212).

XV—MISCELLANEOUS

Visual Purple and the Course of Dark Adaptation.W. A. H. Rushton and R. D. Cohen. *Nature*, 173, 301-302 (13 Feb. 1954).

An apparatus is described in which two different test flashes, travelling by alternate light paths, are compared throughout dark adaptation, and the threshold intensity (logarithmic scale) is plotted against the time from the end of the adaptation. It is concluded that there is no detectable change in the level of visual purple in the 100-fold increase of sensitivity which follows the extinction of an ordinary room light, and reasons are presented for the theory that the chief factor in dark adaptation is an increase in the quantum sensitivity of the retina.

J. W. B.

Mechanism of Chemiluminescence in Solution.II—**Oxidation of 3-Aminophthalhydrazide.** T. Bremer. *Bull. Soc. chim. Belg.*, 62, 569-580 (Oct.-Dec. 1953).

Nine elementary processes which could lead to luminescence are considered on the basis that chemiluminescence is the inverse of an elementary photochemical reaction. It is shown that the energy of the emitted quanta result from four combinations of bond dissociation energies; the most frequent are association and exchange processes. In solution a blue, violet, or ultraviolet chemiluminescence is due to a reaction involving two free radicals. The particular case of luminol (3-aminophthalhydrazide) is considered, where the mechanism of oxidation by univalent

steps is suggested, in which the luminescent reaction consists in an association of two different radicals; a dismutation is less probable.

H. H. H.

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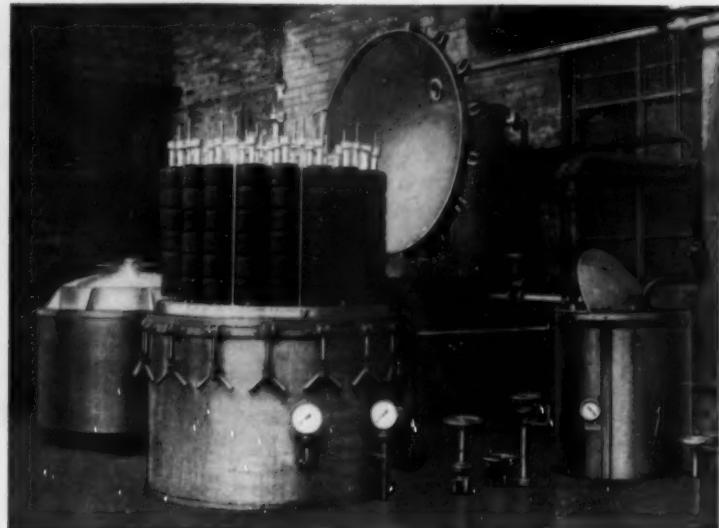
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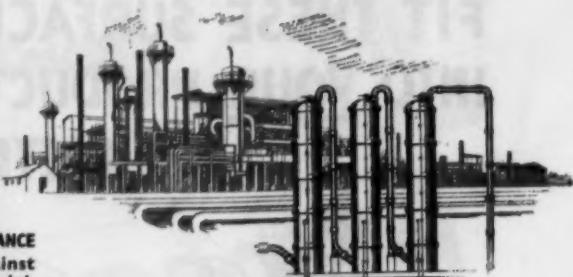
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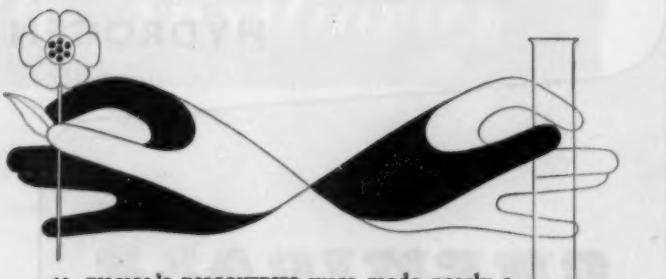
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